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Evaluation of Innovative Combustion Technology for Simultaneous Control of SOx and NOx

Acurex Corp., Research Triangle Park, NC

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National Technical Information Service
NTIS

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April 1987

EVALUATION OF INNOVATIVE COMBUSTION TECHNOLOGY FOR SIMULTANEOUS CONTROL OF SO_x AND NO_x

by

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a. DESCRIPTORS		b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group	
Pollution	Sulfur Dioxide	Pollution Control	13B	07B
Coal	Nitrogen Oxides	Stationary Sources	21D	
Natural Gas	Sorbents	Limestone Injection Mul-		11G
Combustion	Calcium Silicates	tistage Burner (LIMB)	21B	
Limestone	Sodium Carbonates		08G	
Furnaces			13A	

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ABSTRACT

Results are reported on the operation of a 13.8 kW (47300 Btu/hr) Limestone Injection Multistage Burner (LIMB) furnace. Furnace characterization, general sorbent survey and calcium silicate test results are presented. Several candidate sorbent materials for the full scale LIMB demonstration at Edgewater Station in Ohio were evaluated. Measurements of SO_2 capture were made while firing on Pittsburgh No. 8 coal (2.6% sulfur) and natural gas spiked with SO_2. The sorbent injection point was at 1210°C (2210°F). With a furnace quench rate 260°C/s (468°F/s) the average residence time at reaction temperatures was 1.3 s. At molar Ca/S ratios of 2, Vicron 45-3 limestone produced a 38% reduction in SO_2 emissions from baseline levels. Increased SO_2 capture rates, ranging from 60-69%, were measured for various calcitic atmospheric hydrates. Dolomitic sorbents were able to achieve captures of 73 to 75% and showed no significant difference between atmospheric and pressure hydrates. With the addition of sodium bicarbonate ($NaHCO_3$) at molar Ca/Na = 15, captures of 83 and 88% were possible for the calcitic and dolomitic hydrates, respectively. Sodium bicarbonate was evaluated as a fuel additive as well as a sorbent additive.

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CONVERSION FACTORS

To convert from	To	Multiply by
British thermal unit	gram calories	252
degrees Fahrenheit-32	degrees Centigrade	0.5555
feet	centimeters	30.48
cubic feet	cubic meters	0.02832
feet per minute	centimeters per second	0.508
cubic feet per minute	cubic meters per second	0.000472
gallons	liters	3.785
gallons per minute	liters per second	0.06308
grains per cubic foot	grams per cubic meters	2.228
inches	centimeters	2.54
megawatt (electric)	standard cubic feet per minute	0.000535
pounds	kilograms	0.4536
tons (short)	metric tons	0.90718
tons per hour	kilograms per second	0.252

SECTION 1

INTRODUCTION

The environmental impact of nitrogen and sulfur oxides (NO_x and SO_x) emissions from fossil fuel power stations has been an area of major concern. Because of the possible link between these emissions and environmental problems such as acid rain, the U.S. Environmental Protection Agency (EPA) has been involved in developmental studies of in-furnace control technologies. the reduction of NO_x emissions through staged burner systems has been developed and demonstrated in the field. One method to achieve simultaneous reductions in SO_x emissions is the injection of a sorbent material in or just downstream of the flame zone. Because of early efforts in which a staged low NO_x burner was combined with limestone sorbent injection through the burner itself, the technology has been called Limestone Injection Multistage Burner which is abbreviated as LIMB. More recently, the term "LIMB" has become generic and includes in-furnace sorbent injection using materials other than limestone and injection locations somewhat removed from the burners.

As part of EPA's developmental studies into control technologies for SO_x and NO_x emissions, Acurex Corporation was contracted to operate and collect data from the EPA pilot scale Innovative Furnace. Since October 1, 1984, Acurex personnel have provided operation, maintenance, and design support as well as planning, data reduction, and reporting on the results of the experimental program. Operation of the Innovative Furnace by Acurex ended on September 31, 1985. This project final report presents information on the facility design, experimental procedures, fuels and sorbents, and discusses the experimental results.

SECTION 2

EPA INNOVATIVE FURNACE TEST FACILITY

The EPA Innovative Furnace is a down-fired, refractory lined cylinder with an internal diameter of 15.2 cm (6 in.) [Note: EPA officially uses the S.I. units system. See page xiii for conversion factors.] A cross section of the combustion chamber is shown in Figure 2-1. An overall schematic of the furnace and exhaust ducting is given in Figure 2-2. More detailed drawings of furnace sections are included in Appendix A. The facility is nominally rated at 29.6 kw (100,000 Btu/hr, thermal) on either a gaseous fuel or pulverized coal. Burner swirl is fully adjustable and any amount of staging may be accomplished through primary, secondary, and tertiary air flows. The burner design is illustrated in Figure 2-3.

2.1 PILOT SCALE FURNACE

The furnace itself is of conventional construction with rolled steel rings 762 mm (30 in) and 300 mm (1 ft), 450 mm (1.5 ft), or 600 mm (2 ft) high. The steel rings are insulated by four courses of refractory beginning on the outside with a good insulating, low strength castable block mix refractory, progressing inward with castable insulation, following with Castolite 30, and ending on the inside with a high strength, erosion-resistant "hard face" refractory such as Green Cast 97. After casting the refractory into the steel rings, the rings were "stacked" using a gasket made of Kaowool or similar material on the outer periphery. A small amount of uncured refractory was applied around the matching faces of the inner central passage

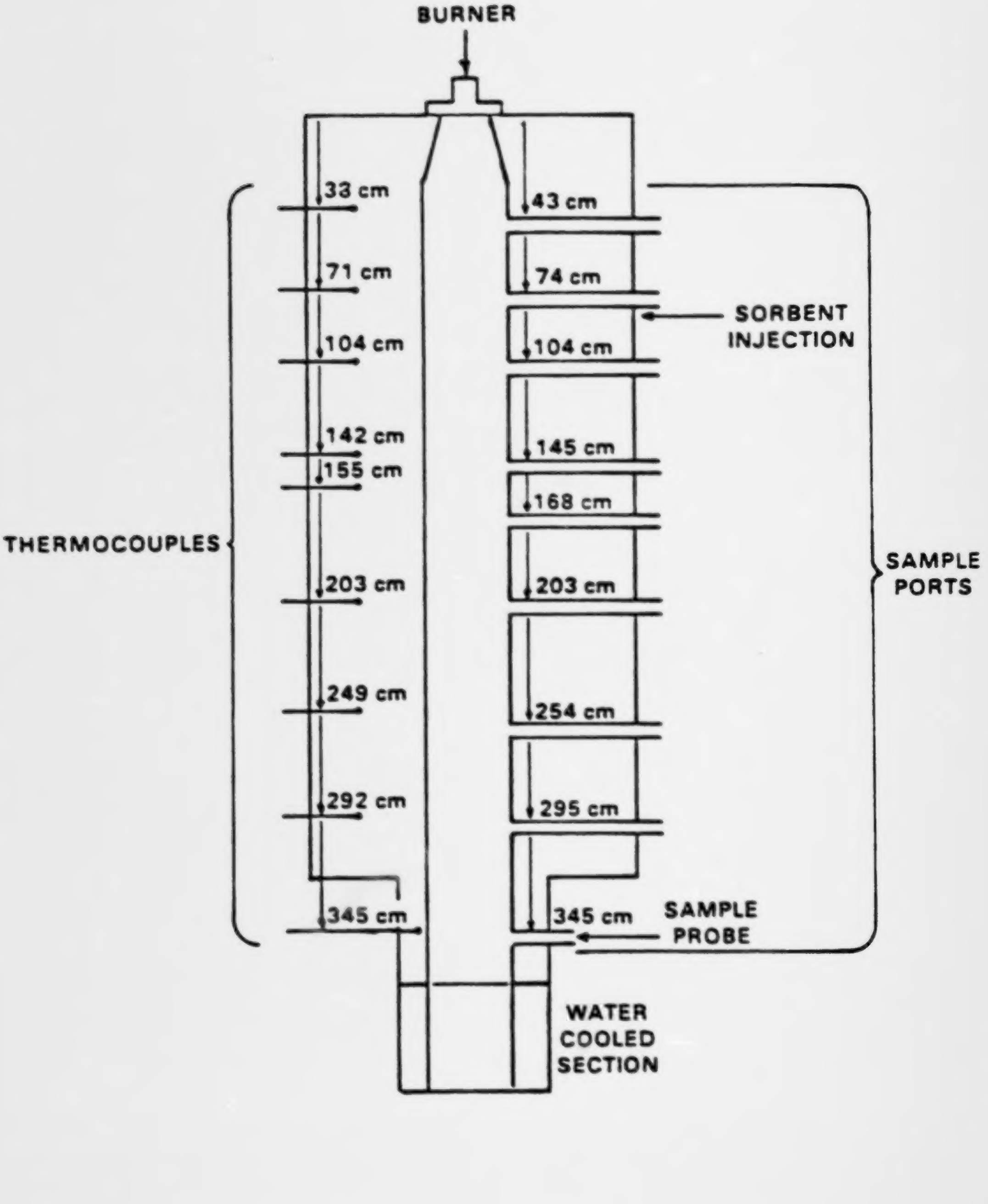
BURNER
THERMOCOUPLES
33 cm
71 cm
104 cm
142 cm
155 cm
203 cm
249 cm
292 cm
345 cm
43 cm
74 cm
104 cm
145 cm
168 cm
203 cm
254 cm
295 cm
345 cm
SORBENT INJECTION
SAMPLE PORTS
SAMPLE PROBE
WATER COOLED SECTION

Figure 2.1. Diagram of EPA Innovative Furnace Showing Locations of Type B Thermocouples, Sampling Ports, Sorbent Injection, and Sample Prove

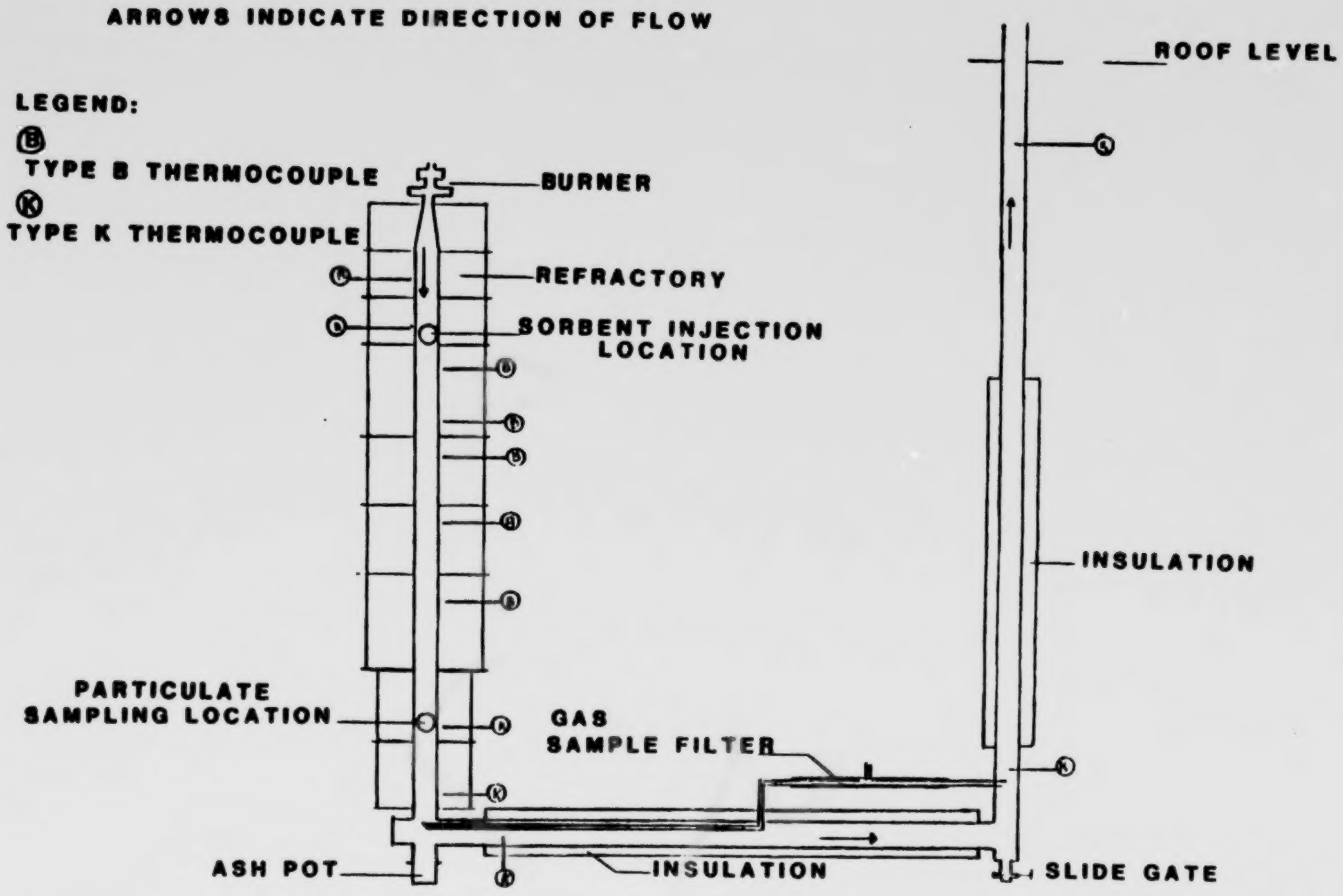
ARROWS INDICATE DIRECTION OF FLOW
LEGEND:
Ⓑ TYPE B THERMOCOUPLE
Ⓚ TYPE K THERMOCOUPLE
BURNER
REFRACTORY
SORBENT INJECTION LOCATION
PARTICULATE SAMPLING LOCATION
GAS SAMPLE FILTER
ASH POT
INSULATION
SLIDE GATE
INSULATION
ROOF LEVEL

Figure 2-2. Schematic of the LIMB Furnace

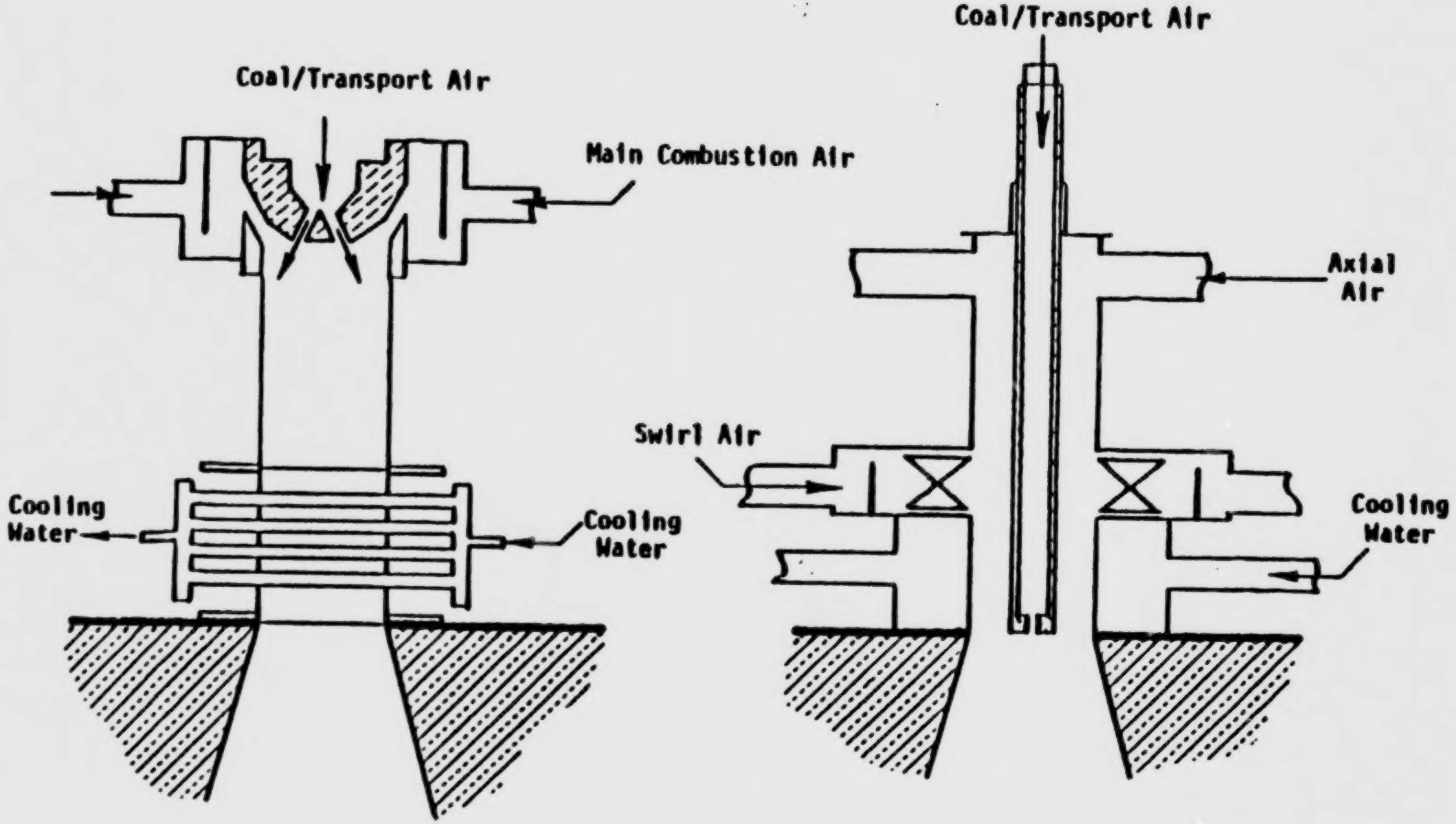
Coal/Transport Air
Main Combustion Air
Cooling Water
Cooling Water
Coal/Transport Air
Axial Air
Swirl Air
Cooling Water

Figure 2-3. Innovative Furnace Burners.

to prevent gas migration to the outer steel shell. Both the gasket and the uncured inner seal material are formed and spread by the weight of the ring and the clamping force of the bolted ring flanges, thus ensuring a good seal at each joint. The joint surfaces of the combustion passage were "faired in" and troweled to give a reasonably smooth and uniform combustion passage.

Inspection or view ports are provided, one a 52 mm (2 in.) circular port at about 190 mm (7 1/2 in.) from the burner tip, and a rectangular port 155 mm x 100 mm (6 in x 4 in.), centered 585 mm (23 in.) below the burner tip. Nine thermocouples, embedded in the refractory approximately 12 mm (1/2 in.) from the combustion gas central passage, are spaced over the 30.48 cm (11 ft) long furnace section. There are nine ports, approximately opposite the thermocouple locations, that are usable for either sampling or injection. Three larger rectangular ports are available for the insertion of chokes and cooling coils to "stage" the combustion or divide the furnace into two or three zones with varied stoichiometry. For example, the first stage can be operated as a "fuel-rich" or "reducing" zone, and the last stage as an air-rich or burnout zone. The final furnace section contains cooling water coils to quench any reactions before the sample is extracted for the continuous monitors.

2.2 SOLID FEED SYSTEMS

Solid fuel, such as coal, is metered from a K-Tron loss-in-weight feeder. A similar feeder, but suited to delivery of a smaller amount, meters the sorbent, such as limestone or lime. The sorbent material is aspirated and transported by air and injected into the furnace through a water-cooled probe. The basic schematics of the coal and sorbent feeders are included in Appendix A. Just beyond the midpoint of the project and prior to the extensive evaluations of the demonstration sorbents, the sorbent feeding system was extensively modified. This was to reduce uncertainties in the sorbent feed due to buildup of sorbent in the transport line and subsequent clogging problems. The approach taken was to minimize dead space volumes and reduce transport line diameters. These changes served to increase flow velocities thereby eliminating, as much as possible, the deposition of sorbent material within the flow system. The increased flow velocity and, therefore, pressure drop in the sorbent transport line and injection probe required the modification of the feeder itself to allow operation of the hopper and entrainment chamber at a slight pressure (3-5 psig). The modifications involved improving the rotating shaft seals and fabricating a new hopper and entrainment chamber. Drawings of the hopper and entrainment chamber are given in Figures 2-4 and 2-5, respectively. A new sorbent injection probe, shown in Figures 2-6 and 2-7, was incorporated into the system. The probe introduces the sorbent flow coaxially to the furnace chamber and provides good sorbent dispersion. As with the sorbent transport lines, it uses 6.35 mm (0.25 in.) of tubing to maintain sorbent flow velocity above 20 m/s (66 ft/s). Smooth bend radii are used to minimize sorbent deposition and clogging.

2.3 INSTRUMENTATION

Operating conditions and emissions were monitored using the following instruments:

Digistrip III Data Logger

Continuous Monitors:

CO	Beckman Model 864 Infrared
CO_2	Beckman Model 864 Infrared
O_2	Beckman Model 755

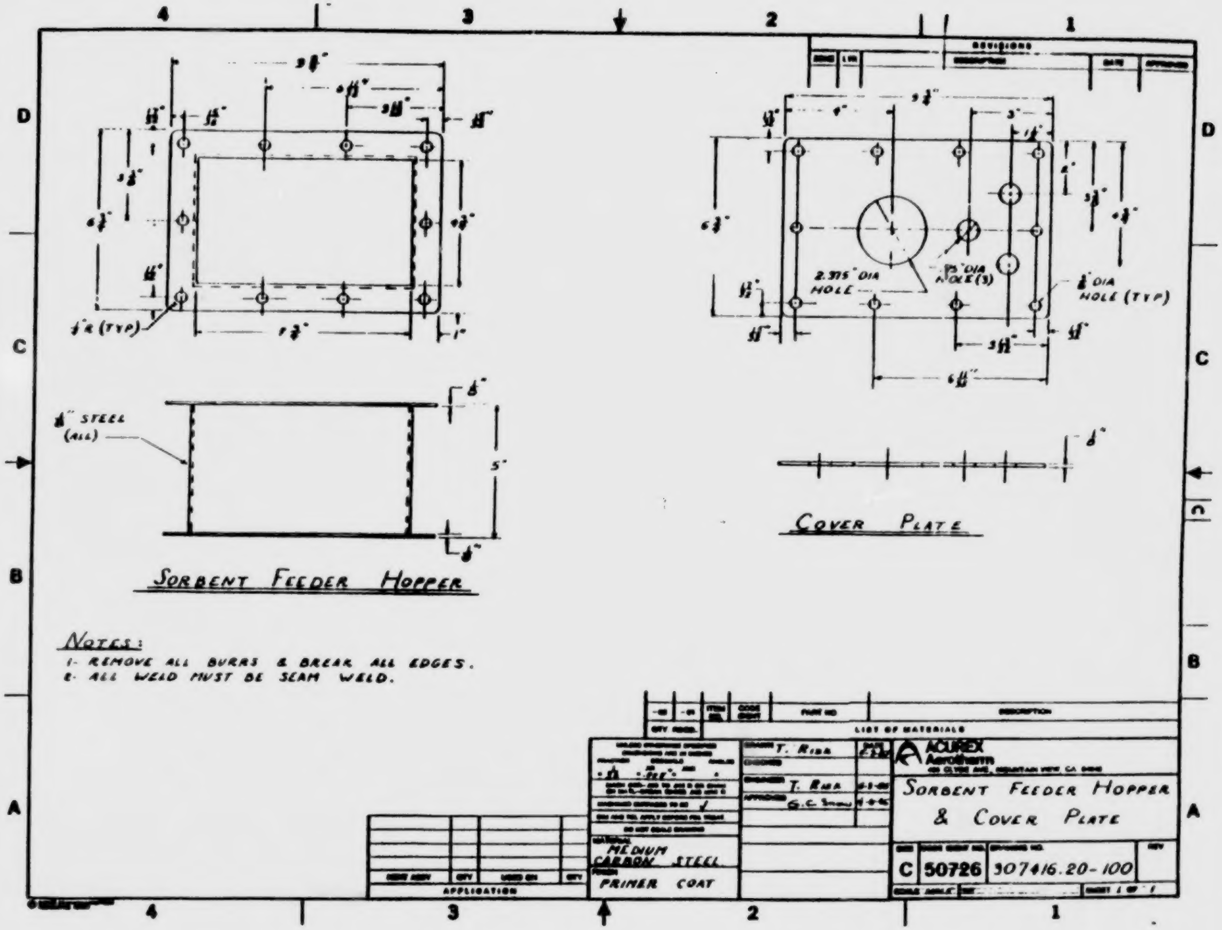
SORBENT FEEDER HOPPER
COVER PLATE
NOTES:
1- REMOVE ALL BURRS & BREAK ALL EDGES.
2- ALL WELD MUST BE SEAM WELD.
STEEL (ALL)
5"
(TYP)
2.375" DIA HOLE
DIA HOLE(3)
DIA HOLE (TYP)
REVISIONS
LIST OF MATERIALS
APPLICATION
ACUREX Aerotherm
SORBENT FEEDER HOPPER & COVER PLATE
T. Riba
G.C. Snow
MEDIUM CARBON STEEL
PRIMER COAT
C
50726
307416.20-100
A
B
C
D
1
2
3
4

Figure 2-4. Sorbent Feeder Hopper

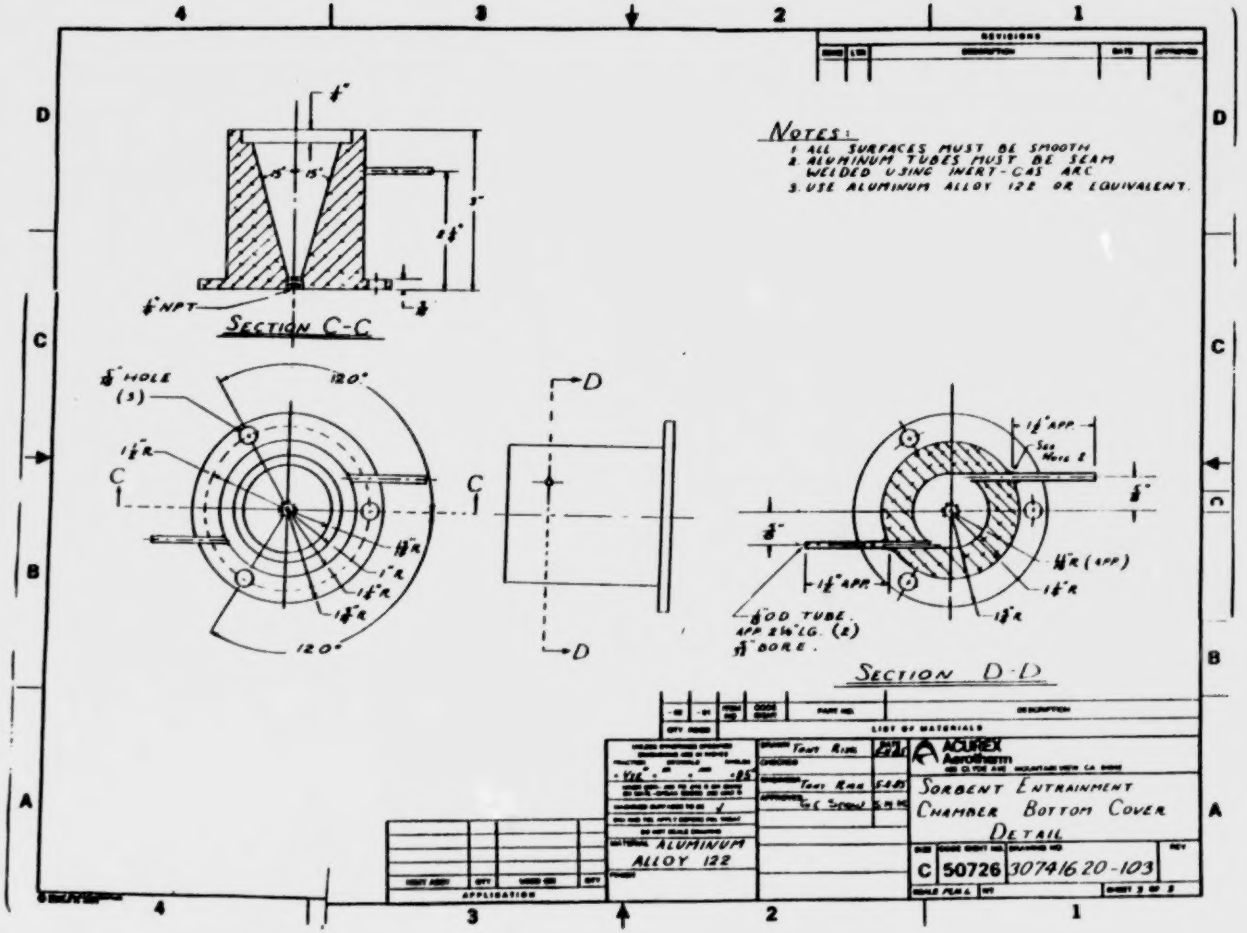
NOTES:
1. ALL SURFACES MUST BE SMOOTH
2. ALUMINUM TUBES MUST BE SEAM WELDED USING INERT-GAS ARC
3. USE ALUMINUM ALLOY 122 OR EQUIVALENT.
SECTION C-C
SECTION D-D
15°
15°
120°
120°
HOLE (3)
NPT
OD TUBE. APP 2¼" LG. (2)
BORE.
See Note 2
C
C
D
D
REVISIONS
LIST OF MATERIALS
ACUREX Aerotherm
SORBENT ENTRAINMENT CHAMBER BOTTOM COVER DETAIL
C
50726
307416 20-103
ALUMINUM ALLOY 122
APPLICATION

Figure 2-5a. Sorbent Entrainment Chamber Bottom Section

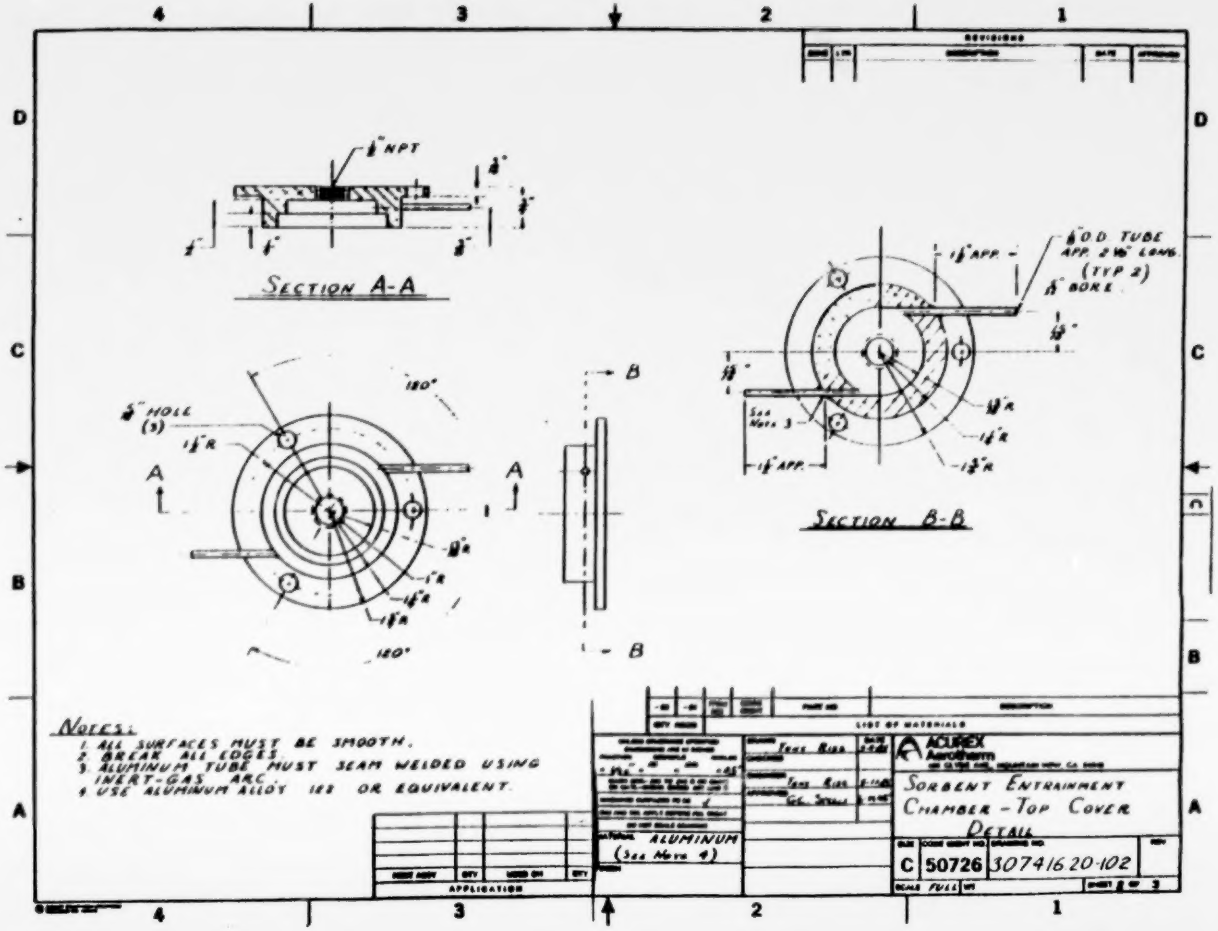
4
3
2
1
D
C
B
A
NPT
SECTION A-A
HOLE (3)
R
120°
A
B
O.D. TUBE APP. LONG (TYP 2)
BORE
APP.
See Note 3
SECTION B-B
NOTES:
1. ALL SURFACES MUST BE SMOOTH.
2. BREAK ALL EDGES.
3. ALUMINUM TUBE MUST SEAM WELDED USING INERT-GAS ARC.
4. USE ALUMINUM ALLOY 122 OR EQUIVALENT.
APPLICATION
LIST OF MATERIALS
MATERIAL ALUMINUM (See Note 4)
ACUREX Aerotherm
SORBENT ENTRAINMENT CHAMBER - TOP COVER DETAIL
C
50726
307416.20-102
SCALE FULL
SHEET 2 OF 3

Figure 2-5b. Sorbent Entrainment Chamber Top Section

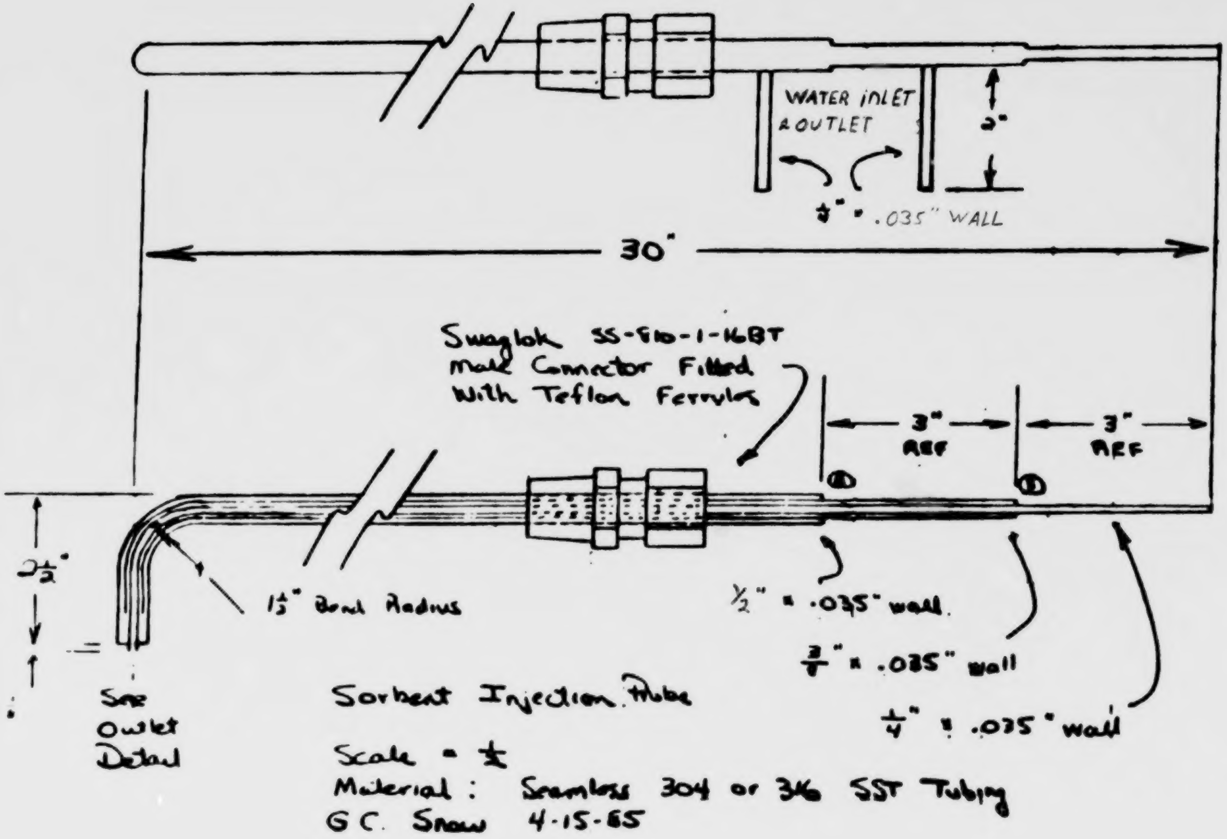
WATER INLET & OUTLET
2"
1/4" × .035" WALL
30"
Swaglok SS-810-1-16BT Male Connector Filled With Teflon Ferrules
3" REF
3" REF
①
①
2 1/2"
1 1/2" Bend Radius
1/2" × .035" wall
3/8" × .035" wall
1/4" × .035" wall
See Outlet Detail
Sorbent Injection Probe
Scale = 1/2
Material: Seamless 304 or 316 SST Tubing
G.C. Snow 4-15-85

Figure 2-6. Sorbent Injection Probe.

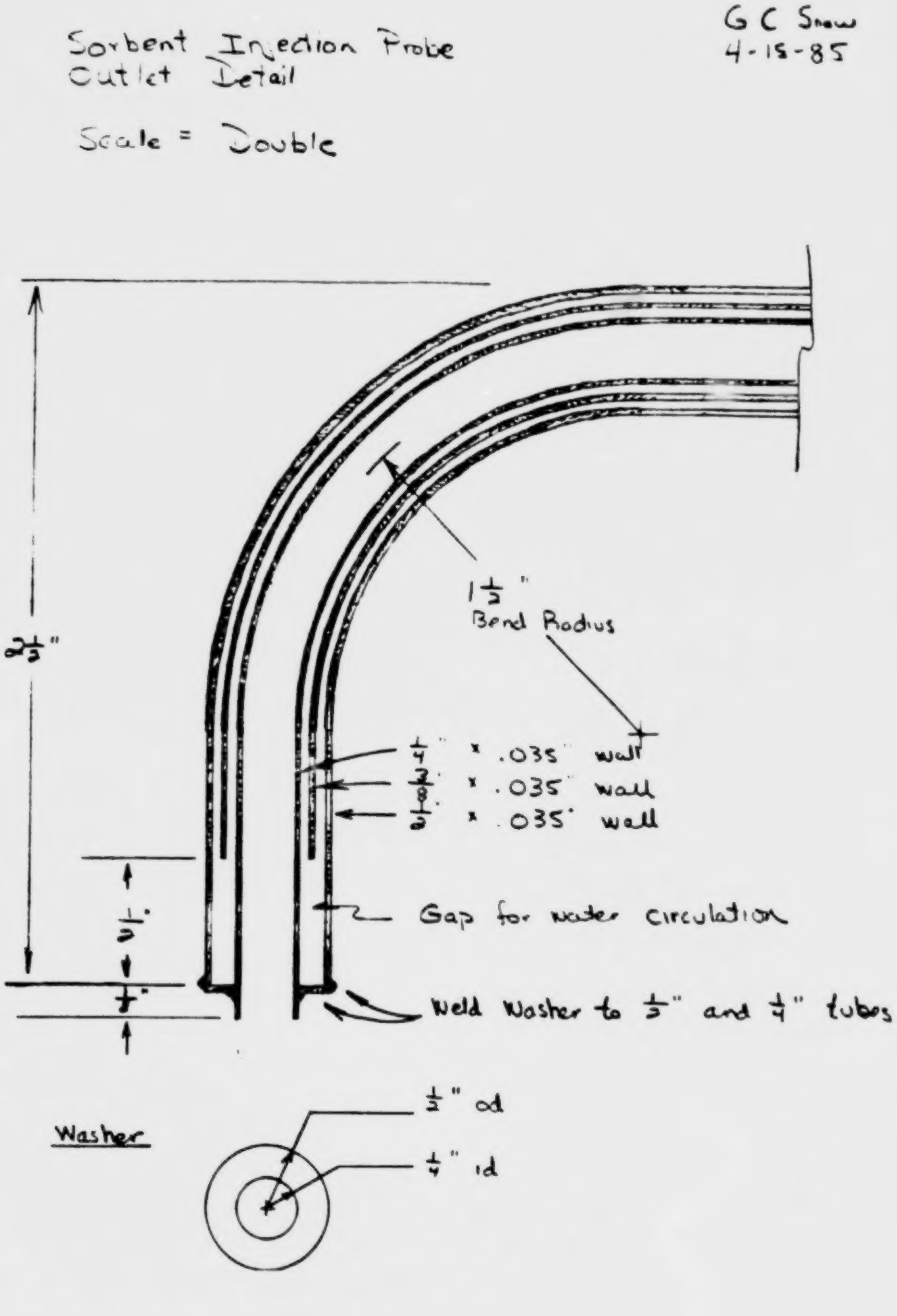
Sorbent Injection Probe
Outlet Detail
G C Snow
4-15-85
Scale = Double
2 1/2"
1 1/2"
Bend Radius
1/4" x .035" wall
3/8" x .035" wall
1/2" x .035" wall
1/2"
1/8"
Gap for water circulation
Weld Washer to 1/2" and 1/4" tubes
Washer
1/2" od
1/4" id

Figure 2-7. Sorbent Injection Probe Detail.

SO_2	Anacon Model 207 U.V.
NO, NO_2, NO_x	Thermoelectron Model 10AR
HC	Beckman Model 402

Thermocouples, both B&K types, embedded or sealed in the refractory.

A schematic of the emission sampling system is given in Figure 2-8. An important feature of the gaseous emission sampling system is the use of an inertial particle filter immediately downstream of the probe. The inertial particle filter draws a side stream of sample gases while the particles continue to flow along the filter tube, eventually returning through an eductor to the stack. This prevents the buildup of a filter cake which could potentially remove SO_2 from the sample gases. The filter and flow lines were heated to a minimum of 120°C (250°F) to prevent condensation. Due to a high rate of gas flow through the filter, its temperature would approach the flue gas temperature, reaching a maximum of 620°C (1150°F).

Sample probe design drawings are included in Appendix A. In addition to the thermocouples fixed in place, a suction pyrometer was used to measure the time/temperature profile in the Innovative Furnace. A drawing of the suction pyrometer design is included in Appendix A.

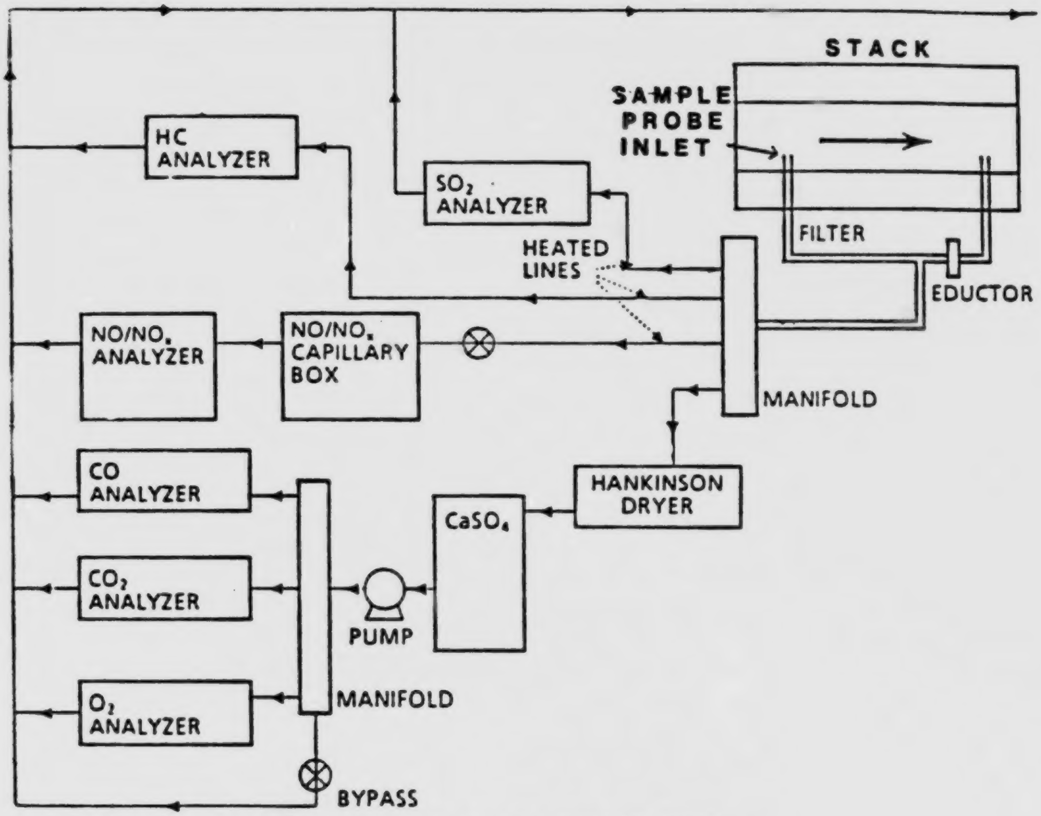
STACK
SAMPLE PROBE INLET
FILTER
EDUCTOR
HC ANALYZER
SO_2 ANALYZER
HEATED LINES
NO/NO_x ANALYZER
NO/NO_x CAPILLARY BOX
MANIFOLD
HANKINSON DRYER
$CaSO_4$
CO ANALYZER
CO_2 ANALYZER
PUMP
O_2 ANALYZER
MANIFOLD
BYPASS

Figure 4- . Continuous Sampling System for the LIMB Furnace.

Arrows indicate direction of sample gas flow.

Figure 2-8. Gaseous Emission Sampling System.

SECTION 3

FUELS AND SORBENTS TESTED

The coals fired in the test program included Pittsburgh No. 8 and Utah bituminous coals and a Montana lignite. The Utah and Montana coals had the lowest sulfur contents at 0.97% and 0.78%, respectively. Pittsburgh No. 8 coal, having an as-received sulfur content of 2.6% by weight, was used as the baseline fuel to screen the candidate demonstration sorbents later in the program. The coal to be used in the full scale demonstration, also a Pittsburgh No. 8, but from the Boich Mining Company, was used late in the test program. Proximate and ultimate analyses of the coals are given in Table 3-1. Detailed elemental analyses of the coals by Inductively Coupled Argon Plasma (ICAP) emission spectroscopy are given in Appendix C. Sulfur capture data were also collected while firing natural gas. To simulate the sulfur content of the Pittsburgh No. 8 coal, SO_2 was added to the natural gas at a concentration of 2.38% by volume. At a combustion stoichiometric ratio of 1.25, the average concentration of SO_2 in the flue gas was 2400 ppm at 0% O_2 giving a close simulation of coal firing.

A large number of sorbents were included in the test program. Table 3-2 lists the properties of sorbents from which test data were collected. Detailed elemental analyses for some of the sorbents are given in Appendix D. Size distribution measurements for the demonstration sorbents are given in Appendix E.

Table 3-1. Coal Analyses

Property	Pittsburgh No. 8	Demonstration Pittsburgh No. 8 Boich Mine	Utah	Montana
Proximate (wt. %, as received)				
Moisture	1.39	2.42	5.57	21.23
Ash	7.51	9.97	9.31	9.34
Volatile Matter	37.23	35.82	39.84	35.16
Fixed Carbon	53.88	51.79	45.28	34.27
Ultimate (wt. %, as received)				
Carbon	74.73	71.65	66.32	53.26
Hydrogen	4.90	4.93	4.78	3.35
Nitrogen	1.86	1.41	1.27	0.87
Chlorine	NR[a]	0.17	0.00	0.01
Sulfur	2.60	3.09	0.97	0.78
Oxygen (by difference)	7.02	6.36	11.78	11.16
Higher Heating Value				
(kJ/kg, as received)	31370	30160	27250	20870
(Btu/lb, as received)	13489	12966	11716	8972

[a] NR = Not Reported

Table 3-2. Sorbent Analyses

Sorbent	Mass Mean Particle Diameter (um)	BET Surface Area (m^2/g)	Chemical Properties (percent by weight)					
			Ca	Mg	Na	H_2O	CO_2	SO_4
Vicron Limestone	11.0	1.01	39.01	0.49	0.022	BDL[a]	43.6	0.04
Fisher Technical Grade $Ca(OH)_2$	NR[e]	12.8	55.0	0.26	BDL	23.2	1.2	BDL
PC III Calcium Silicate	NR	12.0	36.9	NR	NR	6.4	11.6	BDL
Grantsville DPH[d]	NR	24.7	34.0	23.2	BDL	24.2	2.4	BDL
Henderson DPH	NR	16.5	31.5	21.4	BDL	24.5	1.8	BDL
South Western Public Utilities CAH	NR	NR	57.3	NR	NR	NR	NR	NR
Mercer CAH[b]	7.10	17.8	50.5	0.4	0.098	22.0	BDL	BDL
Kemikal CAH	3.88	18.0	49.0	1.0	0.075	23.2	1.2	BDL
Marblehead CAH	7.79	14.3	50.3	0.49	0.01	21.6	1.2	0.13
Detroit Lime CAH	8.33	14.9	50.2	0.51	0.02	22.4	0.4	0.21
Black River CAH	5.53	13.3	49.7	1.6	0.01	21.6	BDL	BDL
Kemidol DAH[c]	19.71	20.6	29.9	18.5	0.17	22.4	3.2	BDL
Ivory Finish DPH	13.72	18.4	28.6	18.1	0.01	23.6	2.8	BDL

[a] BDL = Below Detection Limits
[b] CAH = Calcitic Atmospheric Hydrate
[c] DAH = Dolomitic Atmospheric Hydrate
[d] DPH = Dolomitic Pressure Hydrate
[e] NR = Not reported

Vicron 45-3 was used as a baseline limestone sorbent and Fisher Technical Grade calcium hydroxide was selected as a baseline calcitic hydrate. A sample of calcium silicate (essentially Portland cement), designated as PC III, was also tested in the program. Additional details of the PC III sorbent and test results are given in Section 5.

Additives to the sorbents were also tested to determine their promotional value. Both sodium bicarbonate and sodium carbonate additives were tested. The sodium bicarbonate was a laboratory reagent grade, Fisher number S-233. In a sieve test, 99.7% passed through a 60-mesh sieve. The sodium carbonate was also a laboratory reagent grade, Fisher number S-263. In a sieve test, 100% passed through a 30-mesh sieve and 26.3% passed through a 60-mesh sieve.

SECTION 4

EXPERIMENTAL PROCEDURES

4.1 FACILITY OPERATION

A standardized test plan for the evaluation of sorbents and sorbent/additive mixtures was followed. The plan called for holding the furnace firing conditions constant while varying the sorbent type, feed rate, and injection point as well as the fuel type. A test matrix was devised such that each sorbent would be evaluated under similar conditions according to the standardized test plan.

Before testing a new sorbent, procedures to ensure correct feeding were performed. The feed rates required to obtain Ca/S ratios of 1 and 2 in the combustion zone were calculated from the chemical analysis of the sorbent. The sorbent feeder was recalibrated for each sorbent of different bulk density. If the new sorbent material contained an additive, the sorbent and additive were thoroughly mixed for 30 minutes.

Throughout the study, the furnace firing conditions were held constant. The firing rate was 14 kW (47,300 Btu/hr), the combustion stoichiometric ratio (SR) was 1.25, and the combustion air was preheated to 121°C (250°F). The testing of a sorbent or sorbent/additive mixture was divided into two 1-day test periods. On one day, the sorbent material was injected with the fuel through the burner. On the other day, the sorbent material was injected through a water-cooled probe 81 cm (32 in.) below the burner. The testing routine given below was followed at each sorbent injection point.

1. Switch from natural gas to coal, and adjust air supplies to maintain a combustion SR = 1.25.
2. Insert the sorbent injection probe (if necessary).
3. Calibrate the continuous emission monitors.
4. Ensure stable baseline combustion conditions.
5. Perform a minimum of two sorbent injections at a Ca/S ratio = 1, gathering SO_2 reduction data.
6. Perform a minimum of two sorbent injections at a Ca/S ratio = 2, gathering SO_2 reduction data.
7. Switch from coal to gaseous fuel, and adjust air supplies to maintain SR = 1.25.
8. Ensure proper combustion conditions (without SO_2 doping).
9. Meter SO_2 into the gaseous fuel to provide sulfur loading equivalent to the sulfur content of the coal.
10. Allow SO_2 concentration in flue gas to stabilize.
11. Perform a minimum of two sorbent injections at a Ca/S ratio = 1, gathering SO_2 reduction data.
12. Perform a minimum of two sorbent injections at a Ca/S ratio = 2, gathering SO_2 reduction data.
13. Check the zero and span of the continuous monitors.
14. Shut off SO_2 supply; purge lines.
15. Remove sorbent injection probe (if installed).
16. Reset combustion air supplies for overnight operation.

4.2 DATA COLLECTION AND REDUCTION

A Digistrip III data logger was used to collect experimental data from the continuous emission monitors and the thermocouples. The data logger was programmed to report instantaneous and 2-minute averages of flue gas concentrations. In addition to the data logger, strip chart recorders were used as a visual aid to the operator to ensure stable furnace conditions.

The baseline SO_2 concentration was taken as a 2-minute average when the strip chart recorder indicated relatively stable conditions. The sorbent feeder was started exactly on the data gathering interval. The reduced SO_2 concentration was taken as the first stable 2-minute average after the initiation of sorbent injection. The sorbent injection intervals were as brief as possible to avoid possible effects from buildup on the furnace wall. To ensure the elimination of a possible bias due to wall effects, the SO_2 concentration was required to return to baseline conditions within 10 minutes of the cessation of sorbent feeding for the data to be considered valid. The average response time of the SO_2 analyzer was about 2 minutes. In order to correct for slight variations in furnace conditions, all SO_2 concentrations were corrected to 0% O_2 conditions by Equation (4-1).

$$[SO_2]_{0\%\ O_2} = [SO_2]_m \frac{21}{21 - [O_2]_m} \qquad (4\text{-}1)$$

where

$[SO_2]_{0\%\ O_2}$ = the SO_2 concentration corrected to 0% oxygen concentration in the flue gas

$[SO_2]_m$ = the measured SO_2 concentration

$[O_2]_m$ = the measured O_2 concentration

The corrected SO_2 concentrations and the Ca/S molar ratio are then used to determine the percent calcium utilization by Equation (4-2).

$$\%\ \text{calcium utilization} = \frac{[SO_2]_{\text{baseline}} - [SO_2]_{\text{reduced}}}{[SO_2]_{\text{baseline}}} \times \frac{1}{(Ca/S)} \qquad (4\text{-}2)$$

where

$[SO_2]_{baseline}$ = the baseline SO_2 concentration at 0% oxygen concentration

$[SO_2]_{reduced}$ = the reduced SO_2 concentration during the period of sorbent injection at 0% oxygen concentration

Figure 4-1 is a reproduction of a typical strip chart trace of SO_2 measurements through two sorbent injections. The figure is only for illustrative purposes since experimental data was gathered digitally by the data logger; however, the trace provided a guide to the eye and an indication of equipment performance.

In this case the sorbent utilization was approximately 15% since the change in SO_2 concentration was approximately 30% and the Ca/S ratio is 2.

4.3 QUALITY CONTROL EVALUATION REPORT

The quality assurance objectives of this project were to assure that (1) the combustion conditions in the furnace were representative of the intended experimental design, and that (2) the collected data were accurate and useful. Continuous monitoring methods, as well as particulate sampling from combustion systems, do not permit the collection of true duplicates and spiked samples as in the case of a production analytical chemistry laboratory. Alternative QA/QC procedures required that all continuous monitors and other measurement instruments, where possible, be carefully calibrated using multipoint techniques following manufacturer procedures or other published methods. In addition to the multipoint calibrations, zero and span checks were performed daily. Precision and accuracy performances were checked with calibration standards. These data were recorded into the laboratory notebooks and are archived in the Innovative Furnace laboratory.

SO_2 CONCENTRATION IS TAKEN DIGITALLY
BY KAYE INSTRUMENT

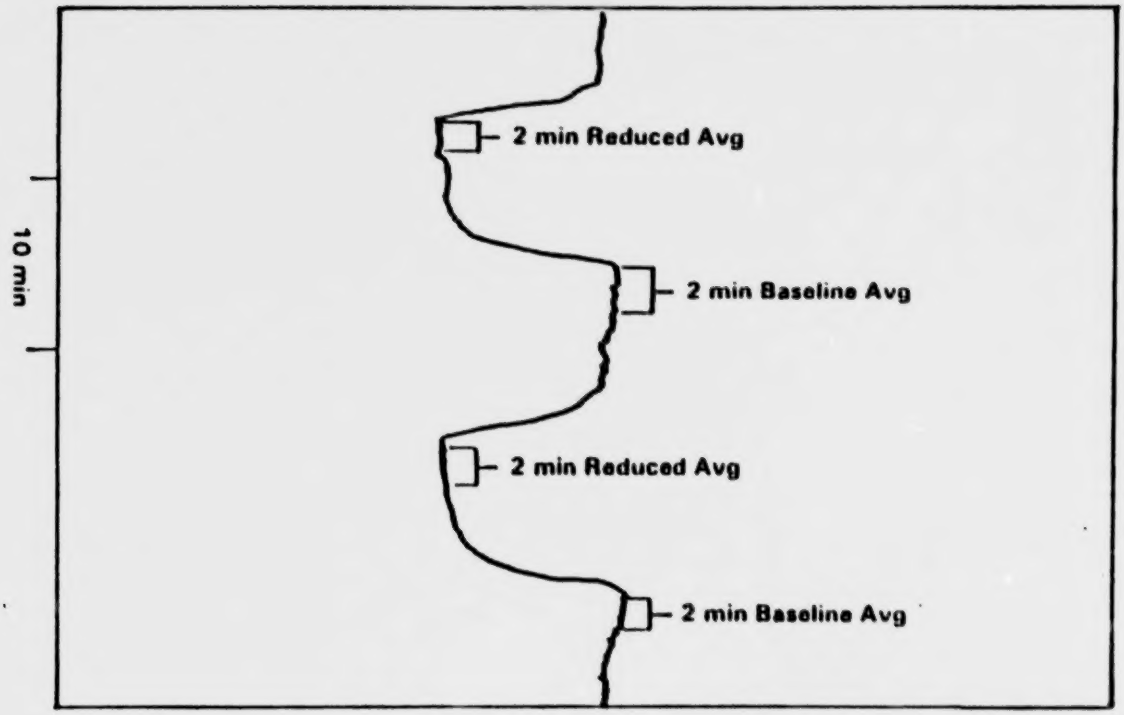

Figure 4-1. Typical SO_2 Strip Chart Trace During Two Sorbent Injections.

4.3.1 Discussion of Data Quality

The major measurements made in the LIMB Innovative Furnace study were coal feed rate, sorbent feed rate, combustion air flow rates, temperature profile in the furnace, and flue gas composition. A summary of the precision, accuracy and completeness achieved in these measurements is listed in Table 4-1 along with the original goals for each type of measurement. In most cases, the completeness, accuracy and precision of measurements made in the Innovative Furnace met or exceeded the quality assurance objectives listed in the Quality Assurance Project Plan (QAPP) developed for the project. The completeness objectives, however, were not met for either the hydrocarbon (HC) or the NO/NO_x concentration measurements. Following instrument operational problems, the project team together with the EPA Project Officer concluded that HC and NO/NO_x measurements were not critical to the project goals and were, therefore, not continued. The accuracy and precision of the coal and sorbent feed rates were determined from the calibration data. As stated in the project QAPP, the gaseous flow rotameters were calibrated at the beginning of the project following both the manufacturer's procedures and the guidelines given in the "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II (EPA-600/4-77-027a). Instrument precision rechecks were not made during the project. However, frequent combustion cross-checks or material balance calculations were performed for each test run to verify continued instrument integrity. Also in keeping with the project QAPP, recalibration of the facility thermocouples were not performed. Instead, the manufacturer's calibration data were accepted and verified by frequent internal cross-checks.

Table 4-1. Data Quality Summary

Parameter (Method)	Completeness (%)		Accuracy (%)		Precision (%)	
	Planned	Actual	Planned	Actual	Planned	Actual
Coal Feed Rate (twin screw feeder)	90	100	±10	±5	±10	2
Limestone Feed Rate (twin screw feeder)	90	100	±10	±5	±10	3
Combustion Oil Flow Rates (rotometer)	90	100	±10	±1[a]	±10	N/A[d]
Temperature (type band K thermocouples)	90	100	±10	N/A[d]	±10	N/A[d]
SO_2 Concentration (ultraviolet)	90	100	±10	±8.0	10/2[c]	1.7/0.7[c]
NO/NO_x Concentration (chemiluminescent)	90	60	±10	±1.4	10/2	5.8/0.7
CO Concentration (NDIR)	90	95	±10	±1.3	10/2	1.2/0.4
CO_2 Concentration (NDIR)	90	100	±10	±6.4	10/2	2.5/0.04
O_2 Concentration (paramagnetic)	90	100	±10	±0.0	10/2	1.3/0.4
HC Concentration (FIA)	90	---	±10	---	10/2	---
Particulate Sampling (pneumatic extractive)	90	100	N/A	N/A	N/A	N/A

[a] Semi-quantitative estimate.

[b] Recalibrations not required by project QAPP.

[c] Two entries are Span Check/Zero Check. Precision values for the zero check are expressed as % of full scale. All other precision values are expressed as % difference using the procedures presented in the project QAPP.

[d] Hydrocarbon Sampling was excluded from project.

A quality control procedure that was constantly employed during the project were material balance cross-checks. The measured concentrations of O_2, CO_2, and SO_2 in the flue gas were compared with theoretically expected values calculated form the coal analysis, firing rate, air flows, and SO_2 flows when added. These material balance calculations were also recorded in the laboratory notebooks. In addition, chemical mass balances are presented in Section 6.4.4.

As specified in the QAPP only internal audits were planned or performed. The results of the internal audit of April 17, 1985 were:

> "Personnel are very familiar with QAPP and QA/QC procedures. Laboratory notebooks are complete and current. The files for sample custody forms and sampling forms are complete and current. Work is proceeding according to QAPP guidelines.

4.3.2 QA/QC Problems Encountered

During the course of the project, it was discovered that an accumulation of ash and slag on the furnace walls would greatly affect the overall experimental results. The effects of wall slagging and the procedural modifications employed to overcome these problems are discussed in Section 4.4. The new operating procedures greatly improved the representativeness and repeatability of the data. Data which had been collected prior to these changes were carefully reviewed. In most cases, the test conditions were repeated to verify or replace the data.

Multipoint calibrations of the Anacon Model 207 Ultraviolet SO_2 monitor showed that the monitor's response curve had become significantly non-linear. Corrective action was required for two purposes: (1) collect valid data in future tests and (2) correct data already collected. Using a curve fitting technique, a mathematical relationship as found to correlate the SO_2 concentration of input gas to analyzer output. The relationship is given by:

$$[SO_2] = 0.695\ [SO_2^*]^{1.04}$$

where $[SO_2]$ = the SO_2 concentration of the calibration gas, ppmv

$[SO_2^*]$ = the instrument response, ppmv

This equation was necessary since electronic relinearization of the Anacon Model 207 was not possible. The loss of linearity in this type of continuous monitor is not uncommon and a correction such as the one above is often employed.

4.4 EFFECT OF FURNACE WALL DEPOSITS

After extended periods of testing, or with long term injection of sorbent, large quantities of ash and partially spent sorbent deposited on the combustor walls. Although a comparison of furnace temperature profiles between operation with and without wall deposits showed very little effect on spatial temperature distribution (see Figures 5-2 and 5-3), the presence and continued buildup of material was undesirable. If continued buildup of the deposits were allowed, flow patterns as well as the temperature profile would undoubtedly be disturbed. In addition, the effects on the residence time of reactants within the temperature window of interest could not be neglected as the combustor volume decreased. Finally, the existence of partially sulfated sorbent on the furnace walls could bias experimental data from run to run.

To determine the impact of these deposits and to uncover other areas of experimental uncertainty, a rigorous test plan was devised to determine precision between successive test runs as well as between alternate days of operation. A test matrix was established in which combinations of two representative coal types (Pittsburgh No. 8 and Utah bituminous) and three sorbents (Vicron limestone, Grantsville DPH, and Fisher Technical grade $Ca(OH)_2$) were tested with a minimum of four data points taken for each set of conditions. From these four data points, precision could be expressed as a 95% confidence limit. By randomly repeating each set of 4 data points on alternate days, the day to day repeatability could be assessed.

Table 4-2 presents the results of this test. The data show that the precision from successive tests within a given day was good and was independent of the magnitude of SO_2 removal. The day to day precision was not so easy to establish. During the testing it was observed that the percent SO_2 removal was dependent on the degree to which the furnace core was fouled with slag and friable wall deposits. These effects can be seen most clearly with the Pittsburgh No. 8/Fisher $Ca(OH)_2$ tests. It was found that with comparing similar amounts of wall fouling the precision was quite good, however, confidence intervals do not intersect. Although it is difficult to quantify the degree of wall fouling and correlations must therefore be qualitative only, the effect of fouling may be illustrated as in Figure 4-2. From this information, new operating procedures were developed to ensure that the furnace walls were clean during data collection. This typically involved data collection on alternate days only, such as Monday, Wednesday, and Friday. On the off days, such as Tuesday, Thursday, and the weekend, the furnace was thermally deslagged using a high natural gas firing rate (about 32 kw or 110,000 Btu/hr) through the main burner. Additionally, an auxiliary natural gas burner "lance" firing about 15 kw (50,000 Btu/hr) was fabricated for use in the furnace mid-section during cleaning operations. Figure 4-3 gives a qualitative illustration of the effect of furnace wall deposits and the improvement due to cleaning or deslagging. The new

Table 4-2. Results of LIMB Pilot Plant Precision Study
All Runs at Ca/S = 2, Sorbent Injected 81 cm Below Burner

			SO$_2$ Removal[b]				SO$_2$[c]
Sorbent/Coal	Date	Furn[a] Wall	N	X	S	95% Confidence Interval	Removal (Percent)
Vicron/Pitts	6/12/85	?	5	29.58	1.96	27.1, 32.0	29.6
	6/21/85	C	5	37.62	1.85	35.32, 39.92	
Vicron/Utah	6/12/85	?	6	26.87	2.99	23.73, 30.01	23.2
	6/24/85	C	5	33.22	2.28	30.39, 36.05	
Grantsville/ Pitts	6/14/85	H	4	51.05	1.74	48.29, 53.81	59.5
	6/18/85	C	4	60.15	1.63	57.56, 62.74	
	6/25/85	C,S	4	62.93	1.24	60.96, 64.90	
Grantsville/ Utah	6/12/85	?	5	48.30	2.52	45.17, 51.42	50.8
	6/25/85	S	3	61.93	1.29	58.73, 65.13	
Fisher/Pitts	6/27/85	S,H	4	42.2	2.44	38.31, 46.08	43.5
	6/19/85	C	4	57.9	1.65	55.27, 60.53	
	6/26/85	S	4	49.0	1.72	46.26, 51.74	
	6/27/85	S	4	51.4	1.73	48.65, 54.15	
Fisher/Utah	6/20/85	S	3	51.03	1.25	47.92, 54.14	23.45
	6/20/85	H	4	44.68	3.55	39.03, 50.33	

[a] Furnace wall condition: C = clean, S = slagged, H = heavily slagged

[b] N = number of repeat data points, X = mean SO$_2$ removal efficiency, S = Standard Deviation

[c] Data collected prior to sorbent feed system upgrade

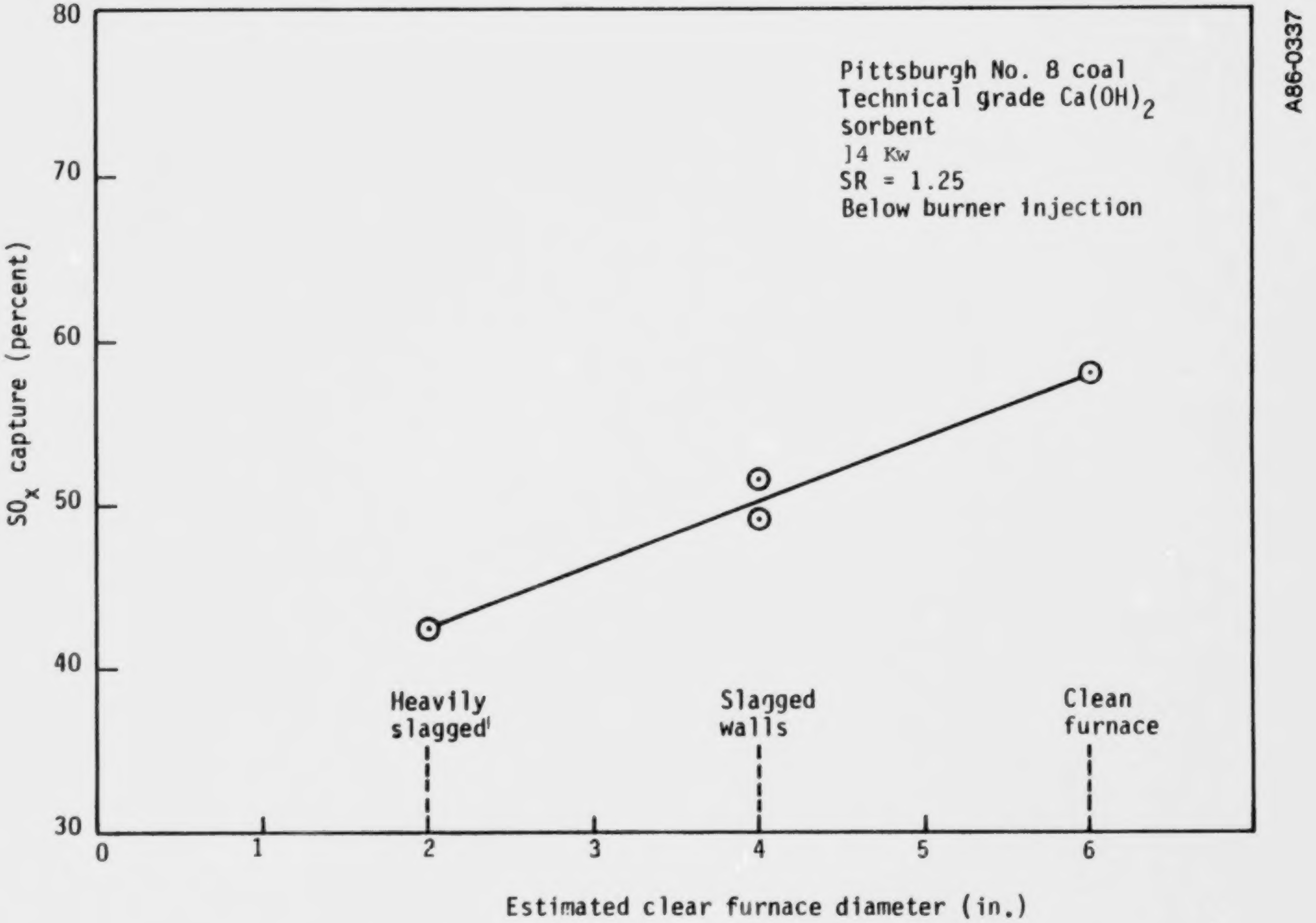

Figure 4-2. Effect of Furnace Wall Deposits on SO_2 Capture.

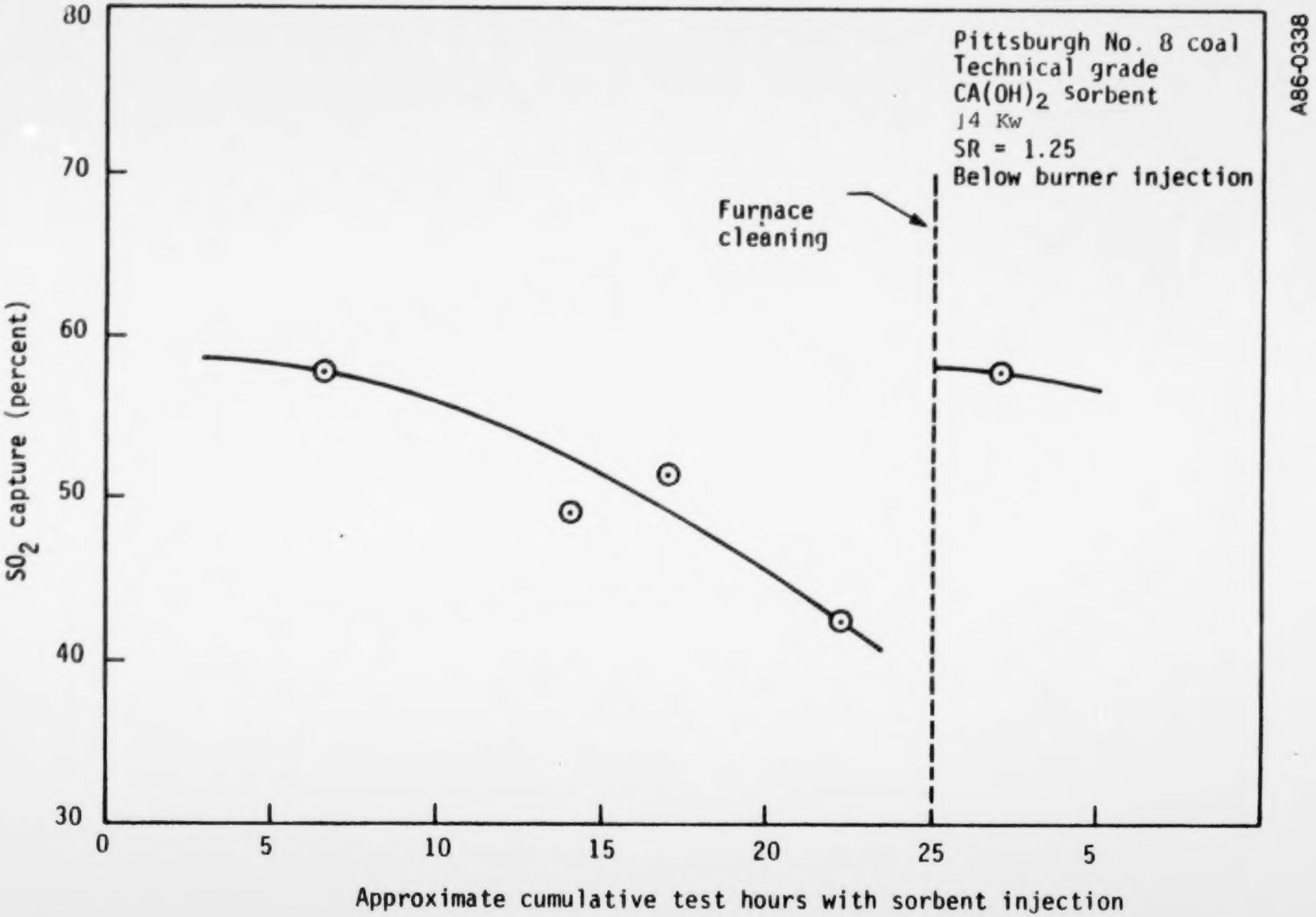

Figure 4-3. Effect of Furnace Cleaning Cycle on SO_2 Capture.

operating procedures together with the modified sorbent feeder and upgraded injection probe (discussed in Section 2.2) resulted in much improved furnace performance and data repeatability.

SECTION 5

EXPERIMENTAL RESULTS AND DISCUSSION

In this section, the test results from the various experimental test matrices are presented and discussed. The results from each major test matrix are presented in respective subsections. In addition to the reduced date shown in tables and plots in the subsections, raw experimental data are included in Appendix B. The first subsection discusses the time/temperature profile as measured in the Innovative Furnace.

5.1 TIME TEMPERATURE PROFILE

A suction pyrometer was used to measure the time/temperature profile in the Innovative Furnace. The profiles for Pittsburgh No. 8 coal and natural gas are plotted in Figure 5-1 as a function of cumulative residence time from the burner. Measurements for natural gas firing taken early in the test program are shown in Tables 5-1 and 5-2. These tables show measurements taken when sorbent air was injected either through the burner or 81 cm (32 in.) below the burner. Figures 5-2 and 5-3 show temperature profile measurements with sorbent air injected at each location and compare clean furnace profiles with those collected when the walls were fouled with deposits. Only when the sorbent air was injected through the burner (Figure 5-3) was there any apparent difference due to furnace fouling.

The sorbent injection point was, for most of the data in this report, 81 cm (32 in.) below the burner. According to Figure 5-1, at this location the furnace temperature was 1210°C (2210°F) during both coal and gas firing. If

Table 5-1. Summary of Suction Pyrometer Testing on EPA Innovative Furnace While Firing Natural Gas at 14.2 kW (47300 Btu/hr) and SR - 1.25 (October 1984)

Distance From Burner	Sorbent Air Injected @ Burner		Sorbent Air Injected 32" Below Burner	
(cm)	Temp °C	Cum. Res. Time (sec)	Temp °C	Com. Res. Time (sec)
73.6	1299	0.528	1324	0.519
104	1219	0.752	1149	0.746
145	1141	1.067	1071	1.077
168	1084	1.253	1024	1.272
203	1016	1.556	961	1.589
254	928	2.016	882	2.068
295	856	2.409	811	2.477
345	744	2.943	722	3.028
Quench Rate (1260-870°C)		230°C/s		254°C/s

Table 5-2. Summary of Suction Pyrometer Testing on EPA Innovative Furnace While Firing Natural Gas at 14.2 kW (47300 Btu/hr) and SR - 1.25 (November 1984)

	Sorbent Air Injection			
	At Burner		32" Below Burner	
Distance From Burner (cm)	Temp (^oC)	Cum. Res. Time (S)	Temp (^oC)	Cum. Res. Time (S)
73.6	1313	0.526	1308	0.528
104	1243	0.749	1171	0.756
145	1123	1.065	1057	1.088
168	1044	1.256	1007	1.286
203	974	1.570	961	1.606
254	887	2.048	884	2.080
295	814	2.458	794	2.494
345	706	3.015	711	3.055

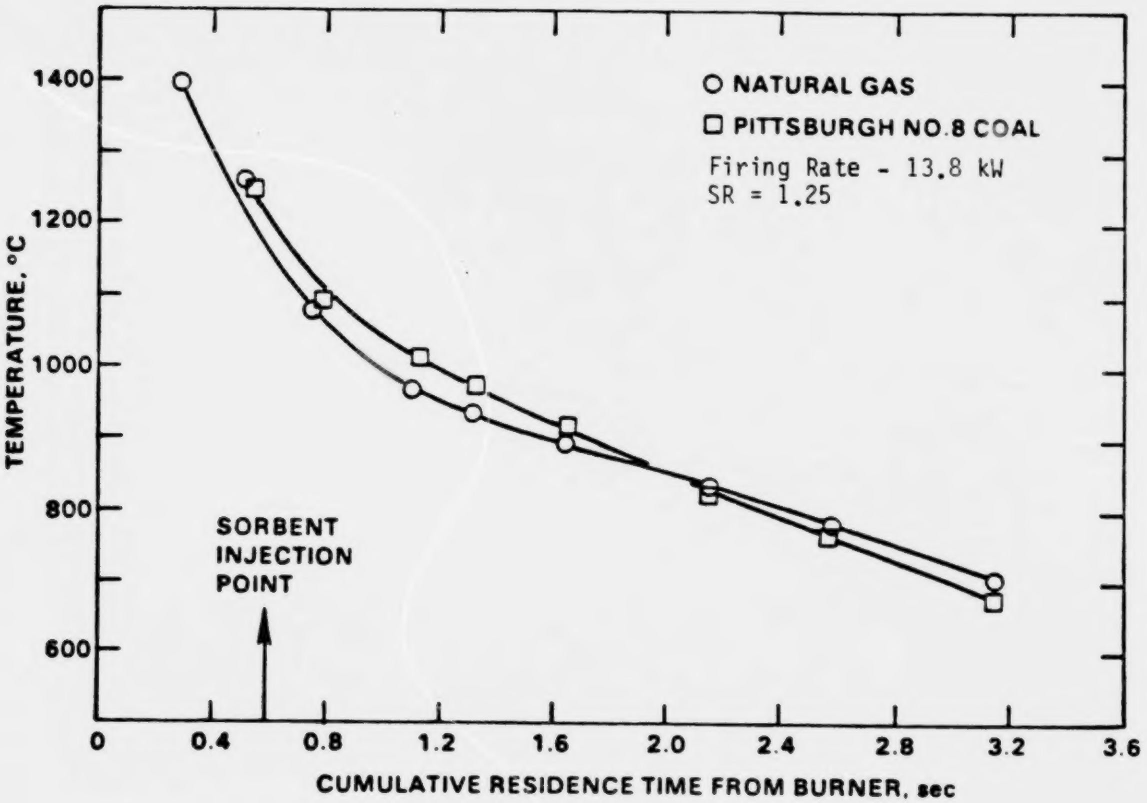

Figure 5-1. Time/Temperature Profile of Innovative Furnace.

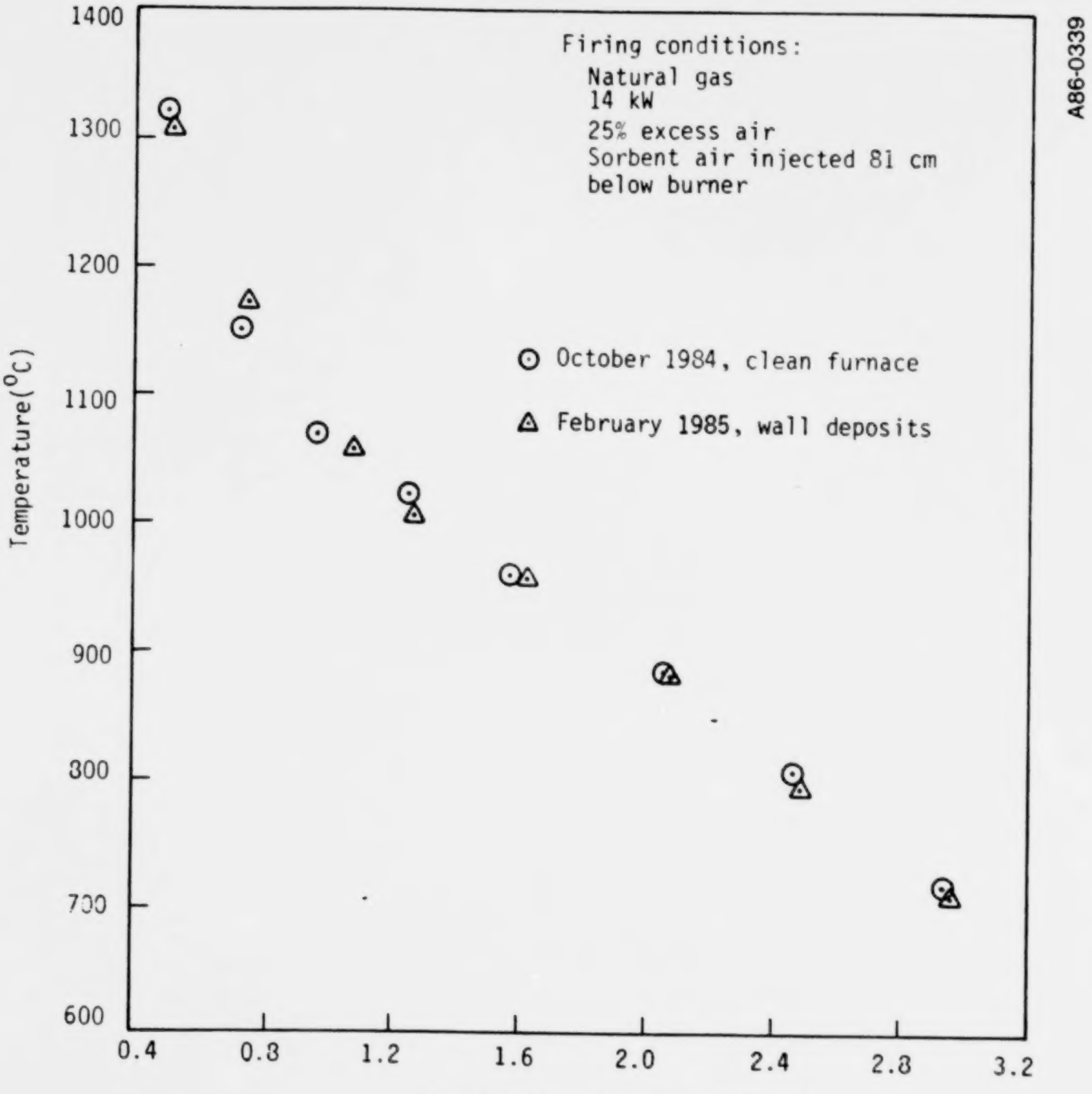

Figure 5-2. Suction Pyrometer Temperature Profile of EPA LIMB Pilot Furnace (Below Burner Injection)

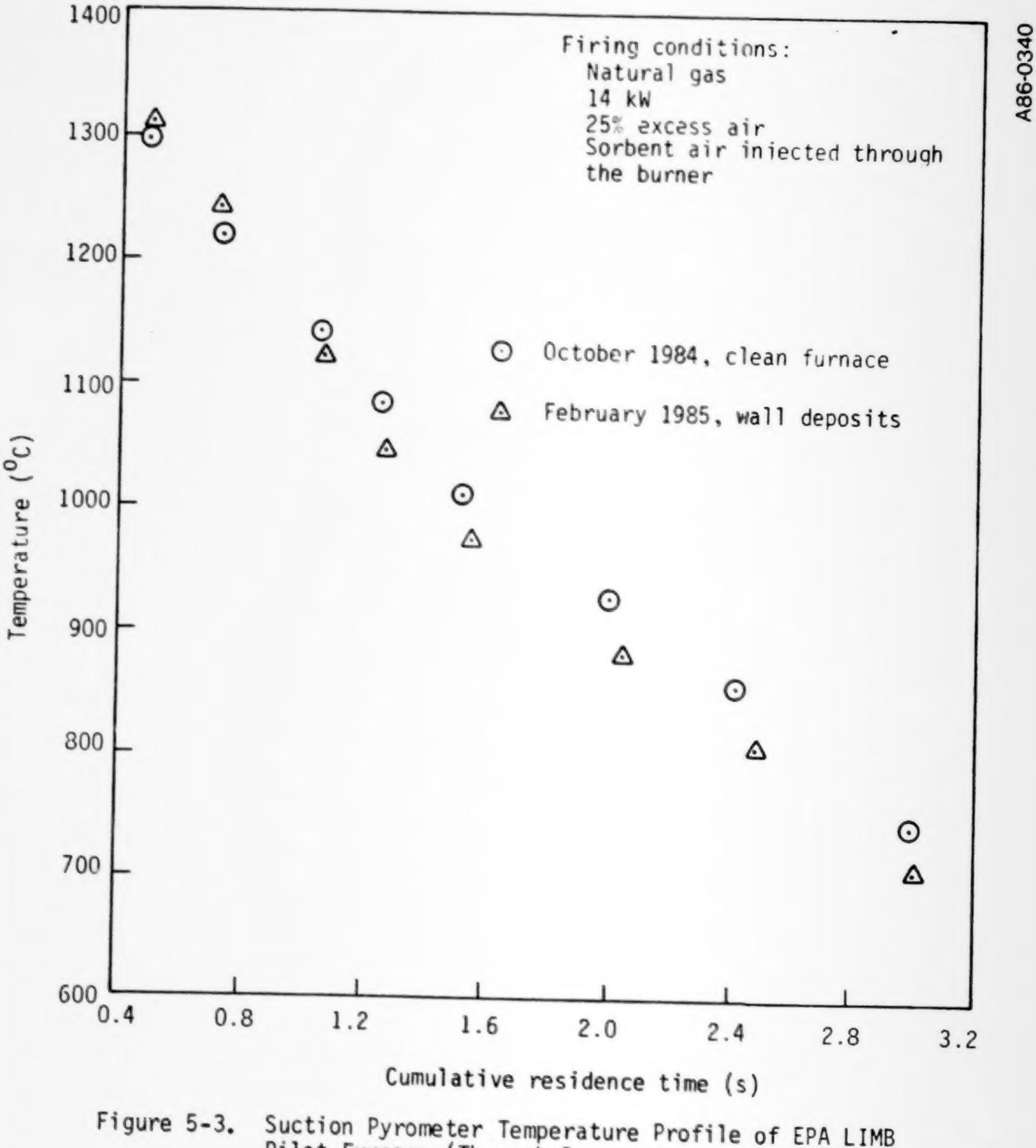

Figure 5-3. Suction Pyrometer Temperature Profile of EPA LIMB Pilot Furnace (Through Burner Injection)

the temperature range of efficient sulfation is assumed to extend down to 870°C (1600°F), then the sorbent experienced a 1.3 s residence time in the reaction window. This gave a nominal quench rate of 260°C/s (468°F/s) for the furnace reaction zone.

5.2 GENERAL SORBENT SURVEY

Early in the test program, several sorbents were available for screening tests and were evaluated in a large test matrix with Pittsburgh No. 8, Utah, and Montana coals as well as with natural gas simulations. The results of this general survey are shown in Table 5-3. On average, the Vicron limestone captured about 38% of the baseline SO_2 emissions while the Fisher Technical Grade $Ca(OH)_2$ and the apparently aged dolomitic hydrates captured about 60%. When a fresh batch of the Henderson dolomitic pressure hydrate was tested, the capture was greater than 80%. The performance of the South Western Public Utilities calcitic atmospheric hydrate was similar to the limestone at 38% on coal. In general, there were only small performance differences between coals. Testing with natural gas gave reasonable agreements with coal firing indicating no major inherent promotional or inhibition effects with coal. Not shown are the effects of variable Ca/S ratios. For each sorbent and fuel combination, a near identical 45% (relative) decrease was experienced in SO_2 capture efficiency as the Ca/S ratio was reduced from 2 to 1 on a molar basis. A few tests of sodium carbonate and sodium bicarbonate additives were also conducted. In general, a 15 to 20% (relative) increase in capture efficiency was seen using these additives. These effects were studied in greater detail with the demonstration sorbents and the results are discussed in Section 5.4.

Spent sorbent samples were collected using the solids sampling probe for many of the sorbents examined in the general survey. The sorbent samples

Table 5-3. Summary of SO_2 Capture Performance on Various Fuels and Sorbents
(Percent SO_2 Removal from Baseline Operation)
Ca/S = 2 molar, no additives

Fuel	Pittsburgh No. 8		Utah		Montana	
Sorbent	Coal	Natural Gas Simulation	Coal	Natural Gas Simulation	Coal	Natural Gas Simulation
Technical Grade Ca $(OH)_2$	58	46*	48	48*	not tested	not tested
Grantsville P. Hydrated Dolomite	60	47	62	62*	62	67
Henderson P. Hydrated Dolomite	50	43	38	48	48	49
Henderson P. Hydrated Dolomite (Fresh Batch)	81	not tested	83	not tested	not tested	not tested
Vicron Limestone	38	36*	33	26*	39	39*
S. Western Public Utilities A. Hydrated Calcitic			38			

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*Natural gas performance not verified with improved sorbent feeder and new test routine. Entry represents estimate made using earlier coal versus natural gas test results.

were collected while firing natural gas both with and without (Ca/S = infinity) SO_2 doping. No sorbent samples were withdrawn while firing coal. The sorbent samples were collected during 10 minute sampling periods and were captured on a heated fiberglass filter. Details of the sampling procedure are given in Appendix A. The collected sorbent samples were analyzed for calcium and sulfate content to determine their degree of sulfation (utilization percentage). Samples collected without SO_2 doping were subjected to BET analysis to characterize their calcined surface area.

The particulate sample analysis data for each of the general survey sorbents while firing on natural gas doped with SO_2 to simulate Utah Coal are summarized in Tables 5-4 through 5-8. The results are expressed in terms of utilization percentage and surface area (S.A.) under the sample analysis column. This allows comparison with the utilization percentage calculated from the Ca/S molar ratio and the percent SO_2 removal as measured by the gaseous emission monitors. The spent sorbent analysis data for the Montana coal simulation are given in Tables 5-9 through 5-11 and for the Pittsburgh No. 8 coal simulation in Table 5-12. Only Grantsville dolomitic pressure hydrate samples were collected for the Pittsburgh No. 8 simulation.

In many cases, the percent sorbent utilization determined from the solid sample analysis was somewhat higher than that determined from the gaseous emission monitors. This may have been due to continued sulfation of the sorbent sample within the sample probe and filter. To evaluate the potential of continued sulfation due to continuous contact with flue gas, an experiment was performed with the sample train. Samples of precalcined sorbents; Grantsville DPH, Henderson DPH, and Vicron limestone, all approximately 0.4 gram quantities, were loaded onto the fiberglass filter to simulate a filter

Table 5-4. Natural Gas Testing with Vicron, Summary of Particulate Sample Analyses Results (SO_2 Doping to Simulate Utah Coal)

Sorbent	Ca/S[a]	Monitors		Sample Analysis	
		SO_2 Removal (%)	Util(%)	Util(%)	S.A. (m^2/g)
Vicron	∞	-	-	16.9	9.13
	1	9.9	9.9	21.7	4.29
	2	18.9	9.5	19.6	3.95
Vicron w/5% Na_2CO_3	∞	-	-	6.8	7.09
	1	7.4	7.4	13.8	5.22
	2	15.9	8.0	22.1	4.00
Vicron w/5% $NaHCO_3$	∞	-	-	8.0	6.25
	1	9.2	9.2	26.9	3.36
	2	15.3	7.7	21.0	3.39

[a] Ca/S = ∞ : Natural gas firing without SO_2 doping

Table 5-5. Natural Gas Testing with $Ca(OH)_2$, Summary of Particulate Sample Analysis Results (SO_2 Doping to Simulate Utah Coal)

		Monitors		Sample Analysis	
Sorbent	Ca/S[a]	SO_2 Removal (%)	Util(%)	Util(%)	S.A. (m^2/g)
$Ca(OH)_2$	∞	-	-	14.2	9.12
	1	12	12	24.6	5.11
	2	25	12.5	19.5	5.96
$Ca(OH)_2$ w/5% Na_2CO_3	∞	-	-	6.2	7.9
	1	15.5	15.5	29.0	22.1
	2	23.7	11.9	26.1	4.28
$Ca(OH)_2$ w/5% $NaHCO_3$	∞	-	-	4.8	7.78
	1	16.6	16.6	27.6	3.07
	2	25.8	12.9	23.4	3.51

[a] Ca/S = ∞ : Natural gas firing without SO_2 doping

Table 5-6. Natural Gas Testing with Grantsville DPH, Summary of Particulate Sample Analysis Results (SO_2 Doping to Simulate Utah Coal)

Sorbent	Ca/S[a]	Monitors		Sample Analysis	
		SO_2 Removal (%)	Util(%)	Util(%)	S.A. (m^2/g)
Genstar-Grantsville	-	-	-	3.4	18.75
	1	27.7	27.7	35.8	8.25
	2	51.5	25.8	38.1	10.88
Genstar-Grantsville w/5% $NaHCO_3$	-	-	-	4.1	17.0
	1	28.1	28.1	58.3	5.65
	2	49.6	24.8	35.3	7.95
Genstar-Grantsville w/5% Na_2CO_3	-	-	-	3.1	16.7
	1	25.7	25.7	59.2	6.32
	2	39.8	19.9	52.6	11.5

[a] Ca/S = - : Natural gas firing without SO_2 doping

Table 5-7. Natural Gas Testing with Henderson DPH, Summary of Particulate Sample Analysis Results (SO_2 Doping to Simulate Utah Coal)

		Monitors		Sample Analysis	
Sorbent	[a]Ca/S	SO_2 Removal (%)	Util(%)	Util(%)	S.A. (m^2/g)
Henderson	-	-	-	10.7	35.7
	1	19.75	19.75	45.4	13.3
	2	38.8	19.4	32.0	21.3
Henderson w/5% Na_2CO_3	-	-	-	8.7	22.9
	1	23.3	23.3	41.4	11.4
	2	47.9	23.95	32.8	15.1
Henderson w/5% $NaHCO_3$	-	-	-	19.0	15.8
	1	25.15	25.15	12.8	9.33
	2	48.3	24.15	44.5	9.35

[a] Ca/S = - : Natural gas firing without SO_2 doping

Table 5-8. Natural Gas Testing with Southwestern CAH, Summary of Particulate Sample Analysis Results (SO_2 Doping to Simulate Utah Coal)

Sorbent	ᵃCa/S	Monitors		Sample Analysis		
		SO_2 Removal (%)	Util(%)	Util(%)	S.A. (m^2/g)	
Southwestern with NO Additive	∞	-	-	2.2	11.1	As received 15.3 m^2/g
	1	12.9	6.5	27.7	6.7	
	2	21.8	10.9	20.8	8.0	
Southwestern with 5% Na_2CO_3	∞	-	-	2.8	10.4	As received 17.7 m^2/g
	1	7.6	3.8	48.8	5.6	
	2	19.4	9.7	26.8	7.7	
Southwestern with 5% $NaHCO_3$	∞	-	-	10.3	7.97	As received 12.7 m^2/g
	1	8.8	4.4	59.5	5.0	
	2	19.5	9.8	29.1	5.43	

[a] Ca/S = ∞ : Natural gas firingwithout SO_2 doping.

Table 5-9. Summary of Particulate Sample Analysis Results for Operation on Natural Gas Doped with SO_2 to 0.78 Percent Sulfur (Montana Coal Simulation)

Sorbent	[a]Molar Ca/S	Sorbent Surface Area (m^2/g)	Sorbent Utilization Percent	Sorbent Utilization Percent
			Sorbent Analysis	Gaseous Emissions
Technical Grade $Ca(OH)_2$		9.70	8.0	-
	1	7.83	20.9	12.3
	2	6.90	20.2	11.5
Technical Grade $Ca(OH)_2$ with 5% Na_2CO_3		11.6	0	-
	1	8.00	12.5	19.7
	2	6.60	29.0	18.4
Technical Grade $Ca(OH)_2$ with 5% $NaHCO_3$		10.5	0	-
	1	5.62	41.8	21.5
	2	5.14	35.2	18.4
Vicron Limestone		8.73	0	-
	1	5.83	9.3	42.1
	2	6.20	9.1	28.6
Vicron Limestone with 5% Na_2CO_3		6.55	0	-
	1	3.25	23.3	38.1
	2	3.21	21.6	32.0
Vicron Limestone with 5% $NaHCO_3$		4.15	6.3	-
	1	2.35	16.8	42.0
	2	1.81	27.1	28.2

[a] $Ca/S = \infty$: Natural gas firing without SO_2 doping

Table 5-10. Natural Gas Testing with Grantsville DPH, Summary of Particulate Sample Analysis Results (SO_2 Doping to Simulate Montana Coal)

Sorbent	[a]Ca/S	Monitors		Sample Analysis	
		SO_2 Removal (%)	Util(%)	Util(%)	S.A. (m^2/g)
Grantsville	-	-	-	7.9	26.25
	1	42.4	42.4	52.4	9.75
	2	66.9	33.45	38.2	15.48
Grantsville w/5% Na_2CO_3	-	-	-	9.5	17.59
	1	40.63	40.63	47.2	10.48
	2	62.75	31.4	47.8	9.43
Grantsville w/5% $NaHCO_3$	-	-	-	12.2	21.53
	1	40.55	40.55	47.8	13.48
	2	71.75	35.9	53.2	12.35

[a] Ca/S = - : Natural gas firing without SO_2 doping

Table 5-11. Natural Gas Testing with Henderson DPH, Summary of Particulate Sample Analysis Results (SO_2 Doping to Simulate Montana Coal)

		Monitors		Sample Analysis	
Sorbent	[a] Ca/S	SO_2 Removal (%)	Util (%)	Util (%)	S.A. (m^2/g)
Henderson	-	-	-	7.9	24.4
	1	24.9	24.9	41.2	13.0
	2	48.75	24.4	33.8	14.15
Henderson w/5% Na_2CO_3	-	-	-	0	25.35
	1	26.05	26.05	46.6	11.45
	2	52.95	26.5	38.3	12.73
Henderson w/5% $NaHCO_3$	-	-	-	0	28.14
	1	30.45	30.45	32.9	13.34
	2	54.35	27.2	32.8	13.93

[a] Ca/S = - : Natural gas firing without SO_2 doping

Table 5-12. Summary of Grantsville DPH Particulate Sample Analysis Results, Natural Gas Firing with SO_2 Doping to Simulate Pittsburgh No. 8 Coal

Sorbent	Point of Injection	[a]Ca/S	Gas Monitors		Particulate Sample Analysis	
			SO_2 Removal (%)	Util(%)	Util(%)	Surf. Area (m^2/g)
Genstar	@	-	-	-	0	3.06
Grantsville	Burner	2	38.7	19.4	18.3	2.60
	32" Below	-	-	-	0	23.7
	Burner	2	46.8	23.4	34.5	10.1
Grantsville	@	-	-	-	3.0	3.04
w/5% Na_2CO_3	Burner	2	32.9	16.45	14.0	1.99
	32" Below	-	-	-	0	24.0
	Burner	2	64.8	32.4	31.6	10.9
Grantsville	@	-	-	-	10.0	2.56
w/5% $NaHCO_3$	Burner	2	24.6	12.3	27.0	2.54
	32" Below	-	-	-		17.4
	Burner	2	67.5	33.75		8.2

[a] Ca/S = - : Natural gas firing without SO_2 doping

cake collected during normal sampling. While firing on natural gas doped with SO_2 to simulate Pittsburgh No. 8 coal (2400 to 2800 ppm of SO_2 in the flue gas at 0% O2), the probe was inserted to draw flue gases for 10 minutes, the normal sampling interval, as well as for a 30 minute period. Nitrogen was used as a quenching and cooling gas. To be sure that the samples were not being contaminated by residue contained within the filter or the probe, or during sample handling, blanks were prepared using the same procedure but without drawing flue gas. Table 5-13 gives the results of the sulfation experiment. Although the measured collection filter temperature of 110 to 150°C (230 to 300°F) was well below the minimum generally assumed for significant reaction, some sulfation of the samples was found. This may have been due, in part, to the high surface area and thus highly reactive calcines used in the experiment. The calcines were produced in a small fluidized bed calciner at 800°C for 30 minutes giving the high surface areas shown in Table 5-13. The sorbents when calcined in the furnace produced considerably lower surface areas and should not be as reactive in the filter. Nevertheless, it can be seen that further sulfation in the sample system may be possible and may have contributed to the discrepancy in the sorbent utilization values.

5.3 RESULTS OF CALCIUM SILICATE TESTING

As part of the general sorbent survey portion of the test program, a calcium silicate based sorbent was evaluated in the Innovative Furnace. The calcium silicate, essentially Portland cement, had shown promising results in combustion environments typical to fluidized bed boilers. In addition, its apparent resistance to sintering and deadburning made it potentially attractive for LIMB applications.

Table 5-13. Sulfation of Precalcined Sorbents in the Sample Train

Sorbent	Blank	10 min. sample	30 min. sample	Surface Area as Blank (m^2/g)
Precalcined Henderson	0	3.26	12.83	28.8
Precalcined Grantsville	0	11.95	15.69	78.5
Precalcined Vicron	0	4.83	9.90	9.90

5.3.1 Background

Studies by Brookhaven National laboratory (BNL) (Reference 5-1) indicated that a calcium silicate based sorbent showed promise for a 40 to 50% reduction in SO_2 emissions from Fluidized Bed Combustion (FBC) of coal. Commercial grade Portland cement provided the source of the calcium silicate. For use in FBC applications, BNL treated the Portland cement to give favorable physical characteristics and a suitable size distribution. This involved hydrating and agglomerating the cement in a drum type pelletizer to form particles in a 16 to 20 mesh size range. These were subsequently cured and dried into hard durable pellets for direct use in the FBC. For bench scale testing in a small fluidized bed reactor, the sorbent was prepared to a size range of 20 to 200 mesh.

BNL's tests indicated that Portland cement Type III (PC III) provided the best performance with respect to sulfation efficiency over other types of Portland cement. They found that a sorbent based on the PC III material gave optimum SO_2 removal at a temperature of 1000°C (1832°F) in an FBC environment. Tricalcium silicate (Ca_3SiO_5) forms 73 to 86% of the silicates present in commercial PC III with the remainder being dicalcium silicate (Ca_2SiO_4). Compositional analysis of PC III presented by BNL showed an average total calcium content of 47.7% by weight.

BNL's investigation utilized a small scale fluidized bed operating on simulated flue gases to collect test data on the PC III based sorbents. Their study revealed several features which made the sorbent attractive for FBC use. These include a sorbent utilization rate or a sulfation efficiency of 50 to 55%. It is important to note, however, that these sulfation efficiencies were achieved over the relatively long sorbent residence times encountered in their fluidized bed reactor. That is, time scales of approximately 2 hours as compared to the 1 to 2 seconds normally available in the reaction "window" (ca. 850-1300°C) of the EPA LIMB Pilot Furnace. The sulfated sorbent demonstrated an ability to regenerate to an average of 95% of its original activity through exposure to a gas flow containing 5% CO as a reducing agent. Another feature which is important for FBC applications was the resistance of the sorbent particles to abrasive wastage and attrition. Additionally, it was suggested that the calcium silicate material retains its pore structure over a higher temperature range than limestone or dolomite based sorbents. This resistance to sintering or deadburning should provide greater freedom in choosing the sorbent injection location in the furnace flame zone.

5.3.2 Test Conditions In The Innovative Furnace

A quantity of BNL treated PC III sorbent was received for testing in the EPA LIMB Pilot Furnace. A test matrix was drawn up to provide injection of sorbent both through the burner and at a location 81 cm (32 in.) below the burner. Operation on both a high sulfur coal and a low sulfur coal was included as well as natural gas doped with SO_2 to simulate the sulfur emissions. Pittsburgh No. 8 coal with an average sulfur content of 2.6% by weight was chosen as the high sulfur coal and the low sulfur coal was represented by a Montana sub-bituminous containing an average of 0.78% sulfur.

A nominal firing rate of 14 kW (47,000 Btu/hr) was used for both coals requiring a flow of 1.52 kg/hr (3.35 lb/hr) for the Pittsburgh No. 8 coal and 2.38 kg/hr (5.25 lb/hr) for the Montana coal. For both coals and the doped natural gas, the sorbent flow was controlled to provide a constant calcium to sulfur molar ratio of 2 (based on a calcium content of 32.2% in the sorbent as determined by the LIMB Materials Characteristic Laboratory). There were no additives combined with either the sorbent or any of the fuels.

The test procedure involved burning doped natural gas or coal at steady state conditions and comparing SO_2 emissions with and without sorbent injection. Each test run began with the injection of sorbent either through the burner or below the burner while operating on natural gas without the addition of SO_2 dopant. The solids sample probe was then used to collect samples of sorbent which had passed through the furnace radiant section. This initial step was taken so that properties of the calcined but unsulfated sorbent, such as surface area, could be determined. In addition, the presence of residual sulfur could be noted.

5.3.3 Test Results And Discussion

Table 5-14 summarizes the test results at both injection locations for each fuel; Montana and Pittsburgh No. 8 coals and the corresponding doped natural gas simulation. The results are expressed as percent SO_2 reduction as compared to operation without sorbent injection. As can be seen, the SO_2 reductions are well below the 40 to 50% values which, according to the findings of BNL, are achievable in FBC applications. It should be noted that SO_2 reductions of 50% or greater have been measured in the EPA Pilot Furnace using unpromoted calcitic or dolomitic sorbents at calcium to sulfur molar ratios of 2 on both Pittsburgh No. 8 and Montana coals. Indeed, reductions of 50% or greater at these stoichiometries are required for economic feasibility of the LIMB process. Therefore, the PC III sorbent material, as supplied by BNL, was unable to provide a significant reduction in SO_2 emissions from the EPA Pilot Furnace.

Table 5-14. SO_2 Reduction Using BNL Treated Portland Cement Sorbent[a]

Fuel	Sorbent Injection Location	
	@ Burner	81 cm Below Burner
Montana (0.78% S)	BDL[b]	16.9
	BDL	17.2
	AVG = BDL	AVG = 17.1%
Natural Gas Doped to 0.78% S	3.2 3.3	13.6
	3.2 4.5	14.5
	AVG = 3.6%	AVG = 14.5%
Pittsburgh #8 (2.6% S)	1.8 3.8	9.9
	3.7 4.5	9.5
	AVG = 3.5%	AVG = 9.7%
Natural Gas Doped to 2.6% S	2.8	12.3
	3.7	12.6
	5.9	
	AVG = 4.1%	AVG = 12.5%

[a] Values indicated are percent reduction of SO_2 with sorbent injection

[b] BDL = Below Determinable Limits

Various explanations for the poor performance of the PC III sorbent have been considered. Firstly, the calcium content of the material received from BNL was checked and compared with the analysis as presented in Reference 5-1. An Atomic Absorption Analysis (AA) conducted in the LIMB Materials Characterization Laboratory indicated that the sorbent contained 32.2 percent calcium by weight. Several Inductively Coupled Argon Plasma (ICAP) analyses were done by the Technical Support Office of the EPA Air and Energy Engineering Research Laboratory (see Appendix C). These analyses measured an average calcium content of 36.9% in the sorbent. While Table 5-14 these values are lower than the 47.4% reported by BNL, it seems unlikely that this alone is responsible for the minimal sulfur capture in the test. There remains the possibility that the reduced calcium percentage was due to the dilution effect of an impurity which may also act as an inhibitor to the sulfation mechanism.

Probably the greatest difference between the FBC process and the more conventional Pulverized Fuel (PF) fired system represented by the Innovative Furnace lies in the effective residence time of the sorbent within the reaction zone. As stated in the BNL paper (Reference 5-1), the sorbent was exposed to SO_2 bearing gas at temperatures averaging 980°C (1796°F) for periods on the order of 2 hours. However, in the Innovative Furnace, the typical residence time for the sorbent particles within the 850 to 1300°C (1560 to 2400°F) reaction window was approximately 1 to 2 seconds. It should be noted that the BNL experimental data is presented in terms of sorbent sulfation percentage rather than reduction of SO_2 emissions in the flue gas. Another potential difference between the FBC and PF processes is the effective calcium to sulfur ratio in the reaction zone. Whereas identical calcium to sulfur ratios may be used in the feed streams of both systems, the effective ratio may be somewhat higher for the FBC. This is due to an inventory of unsulfated sorbent which may exist in the fluidized bed due to the composition of the starting bed material, and periodic batchwise replenishment as the process continues. Finally, within the fluidized bed, there may be more intimate contact between gases and sorbent as compared to a PF fired unit. This would tend to maintain a higher sorbent utilization in the FBC even while the sorbent reactivity was otherwise low.

While the overall SO_2 reductions were low for the PC III sorbent in the Innovative Furnace, it is interesting to note in Table 5-14 that injection below the burner consistently resulted in higher SO_2 removal than injection through the burner. Tables 5-15 and 5-16 summarize the results of analyses on particle samples collected during pure and doped natural gas simulations of Pittsburgh No. 8 and Montana coals, respectively. A comparison of sorbent surface areas show that in every case, the surface area of the sorbent injected through the burner is lower than that for the sorbent injected below the burner. These trends may suggest that the PC III sorbent is not entirely resistant to sintering and loss of activity when exposed to higher flame zone temperatures.

Tables 5-15 and 5-16 also compare the percentage calcium utilization as calculated from both gaseous emissions measurements and solid sample analysis. The calcium utilizations based on the gaseous emissions measurements are somewhat lower than the values determined from the sorbent sulfate analyses. The reasons for this are not entirely clear. Two possibilities which may be considered are continued sulfation of the sample in the probe, and loss of sorbent through deposition on the furnace walls.

Table 5-15. Summary of Portland Cement Particle Sample Analysis Results for Operation on Natural Gas Doped with SO_2 to 2.6 Percent Sulfur (Pittsburgh No. 8 Simulation)

Sorbent Injection Location	Molar Ca/S[a]	Sorbent Surface Area (m^2/g)	Sorbent Utilization (%) Sorbent Analysis	Sorbent Utilization (%) Gaseous Emissions
Through Burner	∞	2.74	18.8	-
	2	1.39	33.5	-
	2	1.22	44.4	2.05
81 cm Below Burner	∞	4.27	22.4	-
	2	2.10	40.1	6.3

[a] Ca/S = ∞: Natural Gas Without SO_2 Doping

Table 5-16. Summary of Portland Cement Particle Sample Analysis Results for Operation on Natural Gas Doped with SO_2 to 0.78 Percent Sulfur (Pittsburgh No. 8 Simulation)

Sorbent Injection Location	Molar Ca/S[a]	Sorbent Surface Area (m^2/g)	Sorbent Utilization (%)	
			Sorbent Analysis	Gaseous Emissions
Through Burner	∞	2.44	14.4	-
	2	1.68	34.9	1.8
81 cm Below Burner	∞	4.82	6.5	-
	2	2.7	41.4	7.3

[a] Ca/S = ∞: Natural Gas Without SO_2 Doping

Although the sample collected in the filter cake by the solids sample probe should be well below the minimum sulfation temperature, there is a limited potential that further reaction with SO_2 could have occurred.

During the test procedure, unusually large amounts of friable material deposited on the combustor walls. Correspondingly, a reduced amount of sample was collected in the solids probe as compared to testing with other sorbents. Therefore, a percentage of the injected sorbent may have been unavailable to the reaction because of deposition on the furnace wall. If this were occurring, then the calculated sorbent utilization based on the gaseous emissions data would be less than that based on the solids analysis. The reasons for the increased wall deposition of the PC III sorbent in comparison to other tested materials are unclear. Perhaps the PC III sorbent had a greater tendency to become sticky at typical operating temperatures and adhere to the furnace walls. Another reason for this behavior may be in the way the PC III particles follow or depart from the gas flow streamlines near the sorbent injection and sampling probes. More information is required regarding the physical and aerodynamic properties of the sorbent particles before a conclusive evaluation can be made.

5.3.4 Conclusions From The Calcium Silicate Testing

The findings may be summarized as follows:

1) Investigations by Brookhaven National Laboratories have suggested that a calcium silicate sorbent derived from a commercial grade Portland cement (PC III) may perform well in Fluidized Bed Combustion applications.

2) The PC III sorbent as tested in the EPA Pilot Furnace was unable to provide a significant reduction in SO_2 emissions. The performance of

the material was well below other sorbents tested to date.

3) A considerable amount of wall deposition in the Pilot Furnace was noted during the injection of the PC III sorbent material.

5.4 RESULTS OF THE DEMONSTRATION SORBENT TESTING

Several candidate sorbent materials for the full scale Limestone Injection Multistage Burner (LIMB) demonstration at Edgewater Station in Ohio were evaluated in the EPA Innovative Furnace. Measurements of SO_2 capture were made while firing at 13.8kW (47300 Btu/hr) on both a Pittsburgh No. 8 coal (2.6% sulfur) and doped natural gas. The sorbent injection point was at 1210°C (2210°F). With a furnace quench rate of 260°C/s (468°F/s) the average residence time at reaction temperatures was 1.3 s. At molar Ca/S ratios of 2, Vicron 45-3 limestone produced a 38% reduction in SO_2 emissions from baseline levels. Increased SO_2 capture rates, ranging from 60 to 69%, were measured for various calcitic atmospheric hydrates. Dolomitic sorbents were able to achieve captures of 73 to 75% and showed no significant difference between atmospheric and pressure hydrates. With the addition of sodium bicarbonate ($NaHCO_3$) at molar Ca/Na = 15, captures of 83 and 88% were possible for the calcitic and dolomitic hydrates, respectively.

5.4.1 Introduction

Seven commercially available hydrated calcitic and dolomitic lime sorbents were tested for their relative SO_2 capture efficiencies in the EPA Innovative Furnace. Hydrated sorbents were of interest due to promising results in previous laboratory and pilot scale work. Vicron limestone was also tested to provide a baseline for comparison with the hydrates. All of these materials were subjected to screening tests to evaluate their performances at constant furnace conditions using a Pittsburgh No. 8 coal and

SO_2-doped natural gas. The best high calcium hydrate and the dolomitic atmospheric hydrate were further tested over a range of Ca/S ratios and with a promoter to determine their responses to additives. Finally, these two sorbents were tested with the coal to be used in the demonstration at Edgewater Station in Ohio.

5.4.2 Description Of Test Fuels And Demonstration Sorbents

Pittsburgh No. 8 coal, having an as-received sulfur content of 2.6% by weight, was used as the baseline fuel to screen the candidate sorbents. The coal to be used in the full scale demonstration, also a Pittsburgh No. 8, but from the Boich Mining Company, became available as the test series was underway. Two of the sorbents were rerun with the Boich coal following completion of the screening tests. Proximate and ultimate analyses of the coals are given in Table 3-1. The percentages of carbon and hydrogen are quite similar for both fuels but the Boich coal had somewhat higher ash and sulfur contents in both the as-received and as-dried forms. Both coals were pulverized to approximately 70% through 200 mesh. When fired at a nominal stoichiometric ratio of 1.25, the Pittsburgh No. 8 coal produced an SO_2 concentration in the flue gas of about 1900 ppm (2350 ppm at 0% O_2). The Boich coal gave a higher concentration at about 2250 ppm (2790 ppm at 0% O_2). NO_x levels were about 440 ppm (540 ppm at 0% O_2).

Sulfur capture data were also collected while firing natural gas. To simulate the sulfur content of the Pittsburgh No. 8 coal, SO_2 was added to the natural gas at a concentration of 2.38% by volume. At a stoichiometric ratio of 1.25, the average concentration of SO_2 in the flue gas was 1890 ppm (2340 ppm at 0% O_2), nearly matching the coal fired condition.

The seven candidate demonstration sorbents were all commercially produced hydrates. These materials consisted of five calcitic atmospheric hydrates (CAH), a dolomitic atmospheric hydrate (DAH), and a dolomitic pressure hydrate (DPH). Vicron 45-3 was used to provide a comparison against limestone. The sorbent properties are given in Table 3-2. As a group, the calcitic hydrates had fairly similar properties, at least from a macroscopic viewpoint. Thermogravimetric analyses indicated that each of the calcitic materials was at least 96% hydrated. The dolomitic materials were also similar with only small differences between the pressure and atmospheric hydrates. The Ivory finish pressure hydrate showed a greater degree of hydration and a smaller mean particle size than the Kemidol atmospheric hydrate.

5.4.3 Test Conditions

For each test run, the firing rate was held constant at 13.8 kW (47300 Btu/hr, thermal). The air and fuel inputs were targeted to give a nominal stoichiometric ratio of SR = 1.25. However, updated fuel analyses together with the actual control setpoints used resulted in stoichiometric ratios during data collection of SR = 1.24 for the Pittsburgh No. 8 coal and SR = 1.28 for the natural gas. The Boich coal was fired at the target SR = 1.25 condition.

A suction pyrometer was used to measure the time/temperature profile in the Innovative Furnace. This information is plotted in Figure 5-1 as a function of cumulative residence time from the burner. At a constant fuel heat input value of 13.8 kW and the stoichiometric ratio used for each fuel, the temperature profiles are nearly identical. Although minor changes in stoichiometric ratios during data collection will cause small shifts in the furnace temperature profile, the effects were minimal. This was shown during repeat runs in which removal efficiencies of SO_2 remained constant (within random experimental error) while the actual stoichiometric ratio varied by plus or minus 5% from the setpoint value.

The sorbent injection point was 81 cm (31.9 in.) below the burner. At this location, the furnace temperature was 1210°C (2210°F) during both coal and gas firing. If the temperature range of efficient sulfation is assumed to extend down to 870°C (1600°F), then the sorbent experienced a 1.3 s residence time in the reaction window. This gave a nominal quench rate of 260°C/s (468°F/s) for the furnace reaction zone.

5.4.4 Results And Discussion

The SO_2 removal efficiencies of the various sorbents are summarized in Table 5-17 at a constant Ca/S molar ratio of 2. Removal efficiencies range from a low of 38% for Vicron to a high of 75% for the Ivory Finish DPH on Pittsburgh No. 8 coal. The values in the table as well as in subsequent figures represent averages of 4 to 6 data points. The repeatability of the data was very good as characterized by an average standard deviation of 3.2% (relative basis).

In every case, firing on SO_2 doped natural gas gave lower SO_2 removal than coal firing. This is illustrated for Vicron, Marblehead, and Kemidol sorbents in Figure 5-4. The difference is small for Vicron. However, SO_2 removals for both the calcitic and dolomitic hydrates fell significantly during gas firing. The reasons for this behavior are unclear. Inherent promotional effects due to certain components in the coal ash (e.g., Mg, Na, K, Cr) cannot account for the observed difference between the two fuels. One possible explanation may involve the use of a water cooled solids collection probe which

Table 5-17. Summary of SO_2 Capture Performance of Various Sorbents (13.8 kW Firing Rate, 1210°C Injection Temperature)

Sorbent	(Percent SO_2 Removal at Ca/S = 2, Molar) Natural[(a)] Gas	Pittsburgh No. 8 Coal	Demo Coal Pittsburgh No. 8 Boich Mine
Vicron Limestone	36	38	NT[b]
Mercer CAH	53	59	NT
Kemikal CAH	36	62	NT
Marblehead CAH	46	69	69
Detroit Lime CAH	42	60	NT
Black River CAH	42	61	NT
Kemidol DAH	65	73	83
Ivory Finish DPH	67	75	NT

(a) Doped with 2.38% by volume SO_2

[b] NT = Not tested

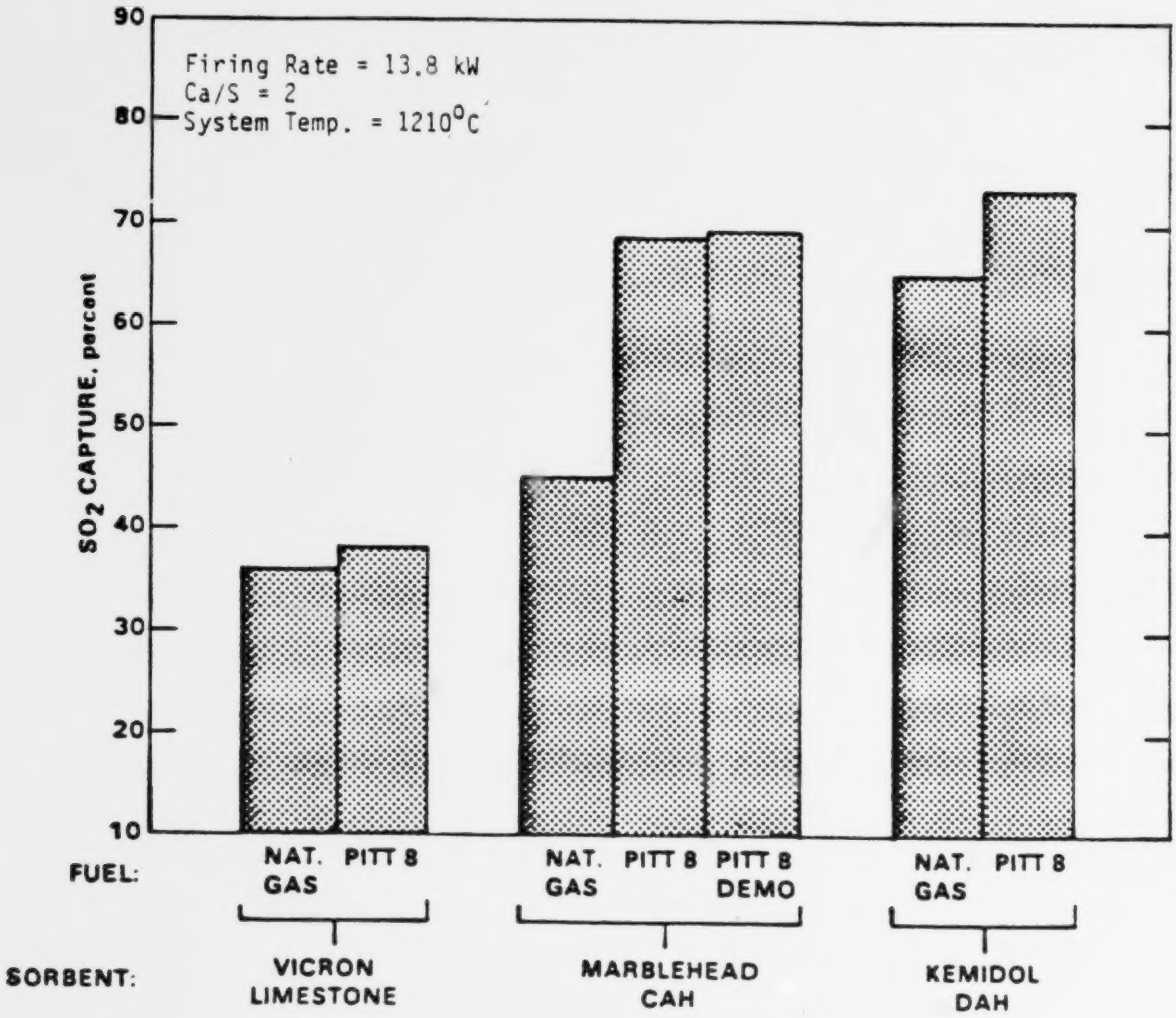


Figure 5-4. Effect of Fuel Type on SO_2 Capture for Vicron Limestone and Marblehead and Kemidol Hydrates.

was inserted into the bottom of the furnace for gas testing but was removed during coal firing. Although this probe was at least 120 cm (47.2 in.) below the reaction zone, the heat sinking effect may have caused a more rapid quench of the flue gases giving an effectively shorter residence time at reaction temperatures.

Unfortunately, the project came to a close before further measurements could be made to reach a conclusive answer.

The relative performances of the sorbents using Pittsburgh No. 8 coal at a Ca/S ratio of 2 are illustrated in bar graph form in Figure 5-5. The Vicron limestone captured 38% of the SO_2 emissions. The hydrated calcitic sorbents produced an average SO_2 removal efficiency of 62%. One of these materials, Marblehead, gave very high capture performance at 69%. For a given sorbent type, correlations between SO_2 capture efficiency and properties of surface area, particle size, or elemental stoichiometries (given in Table 3-2) are not evident. The two dolomitic hydrates were able to achieve sulfur captures of 73% and 75% for the Kemidol and Ivory Finish materials, respectively. Although the pressure hydrated Ivory Finish gave the higher removal efficiency, the difference is considered statistically insignificant as it is on the order of one standard deviation for the dolomitic sorbents. The performances of the three classes of sorbents studied in this program --limestone, calcitic hydrates, and dolomitic hydrates -- have similar rankings to those found in other pilot scale research efforts. The results of Overmoe et al. (5-2), Beittel et al. (5-3), and Bortz and Flament (5-4) are compared with the results of this project in Table 5-18. The data in this table show a clear and relatively consistent relationship between the materials: dolomitic sorbents

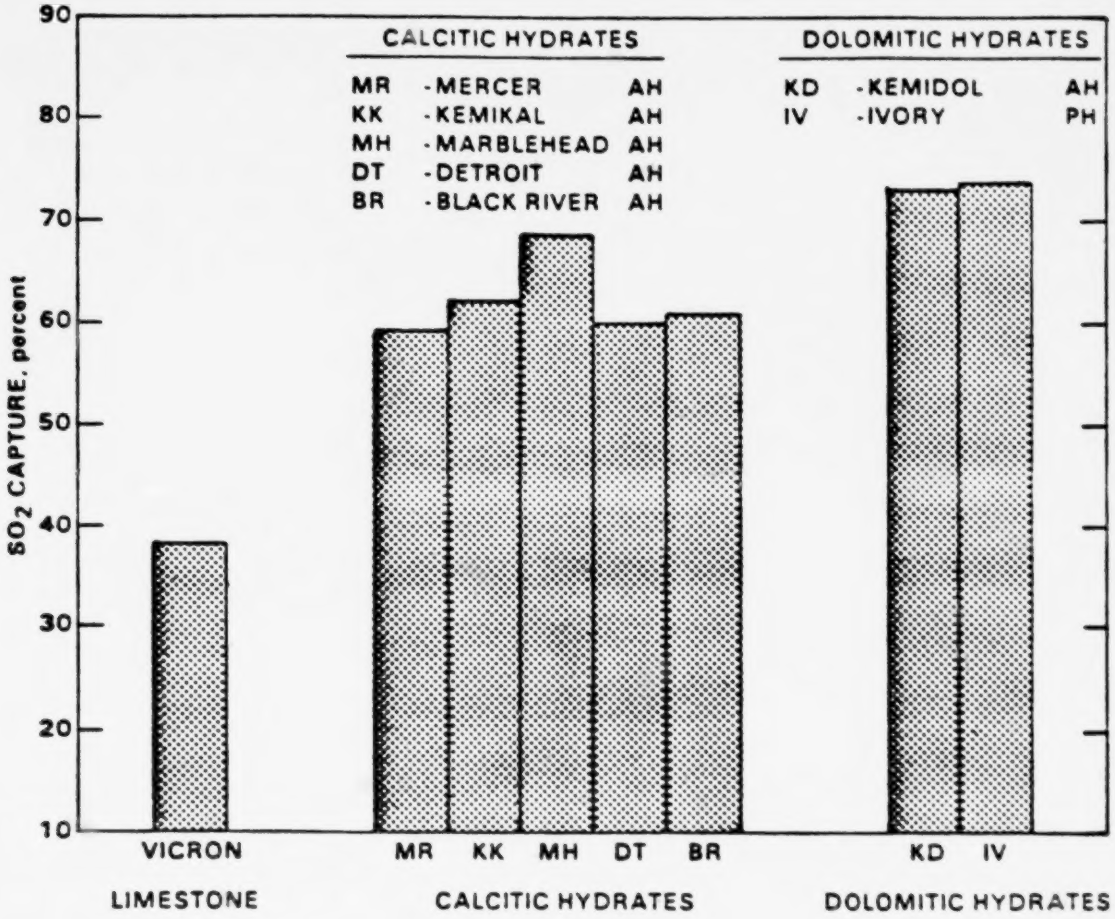


Figure 5-5. Sorbent Performance Summary With Pittsburgh No. 8 Coal at Ca/S = 2, Molar

Table 5-18. Percent SO_2 Captures Achieved with Different Classes of Calcium-Based Sorbents

Reference	Approx. Inj. Temp., °C	Limestone	Calcium Hydroxide	Dolomite	Pressure Hydrated Dolomite
Overmoe et al.(5.2)	1230	30	42 48	57	74
Beittle et al.(5.3)	1200	40	50-60	55	70-80
Bortz & Flament(5.4)	1200	30-40	65-80	--	--
This study	1210	38	59-69	--	75

give increased capture over calcitic sorbents and hydrates are better than carbonates.

Sulfur mass balance calculations were performed for a number of runs where spent sorbent solid samples were collected. The average sulfur balance was greater than 92% indicating that nearly all of the input sulfur was accounted for in the flue gas or fly ash. Table 5-19 summarizes the sulfur balances calculated for each of the demonstration sorbents.

Two of the candidate sorbents, Marblehead and Kemidol, were subjected to further testing with the baseline coal following the screening runs. Figure 5-6 shows the effect of variable Ca/S ratio on capture efficiencies for the two materials. At Ca/S ratios of 2.5, the SO_2 removal rates were quite high at 77% for Marblehead and 80% for Kemidol. The changing slope of the curve fitted to the data illustrates the decreasing calcium utilization at increasing Ca/S ratios. At Ca/S = 1.5, the calcium utilization efficiencies for Marblehead and Kemidol are 37% and 41%, respectively. The utilizations fall to 31% and 32% for the two sorbents at Ca/S = 2.5.

Sodium bicarbonate ($NaHCO_3$) additive was investigated for its promotional effects on the Marblehead and Kemidol sorbents. This reagent was chosen because of its smaller particle size which was expected to give higher reactivity over sodium carbonate (Na_2CO_3). Sieve measurements showed $NaHCO_3$ to be 99.7% less than 250 um compared to 26.3% less than 250 um for Na_2CO_3. Tests were conducted in which the additive was mixed with either the sorbent or the coal to give a Ca/Na ratio of 15. Figure 5-7 shows the test results at a Ca/S ratio of 2 and nominal furnace conditions. Sodium bicarbonate appears to be an effective promoter, increasing possible sulfur captures to 83% and 88% for the calcitic and dolomitic hydrates, respectively.

Table 5-19. Summary of Sulfur Balance Calculations for Demonstration Sorbent Screening Test

Sorbent	Sulfur Balance (%)	
	Input Basis*	Output Basis**
Mercer	94	100
Kemikal	102	91
Marblehead***	100	98
Detroit	100	92
Black River	102	94
Kemidol	78	78
Ivory	86	74
Average	95	90

Firing Conditions: Fuel = natural gas; 47200 Btu/hr
S.R. = 1.25; Ca/S = 2 molar
Sorbent injected 32" below burner

$$* \quad \% \text{ Sulfur Balance} = \frac{\text{Flue gas content (lb/hr) + captured by sorbent (lb/hr)}}{\text{coal input (lb/hr)}} \times 100$$

$$** \quad \% \text{ Sulfur Balance} = \frac{\text{Flue gas output w/sorbent + captured by sorbent (lb/hr)}}{\text{Flue gas output w/o sorbent}} \times 100$$

***Ca/S = 1.86 molar

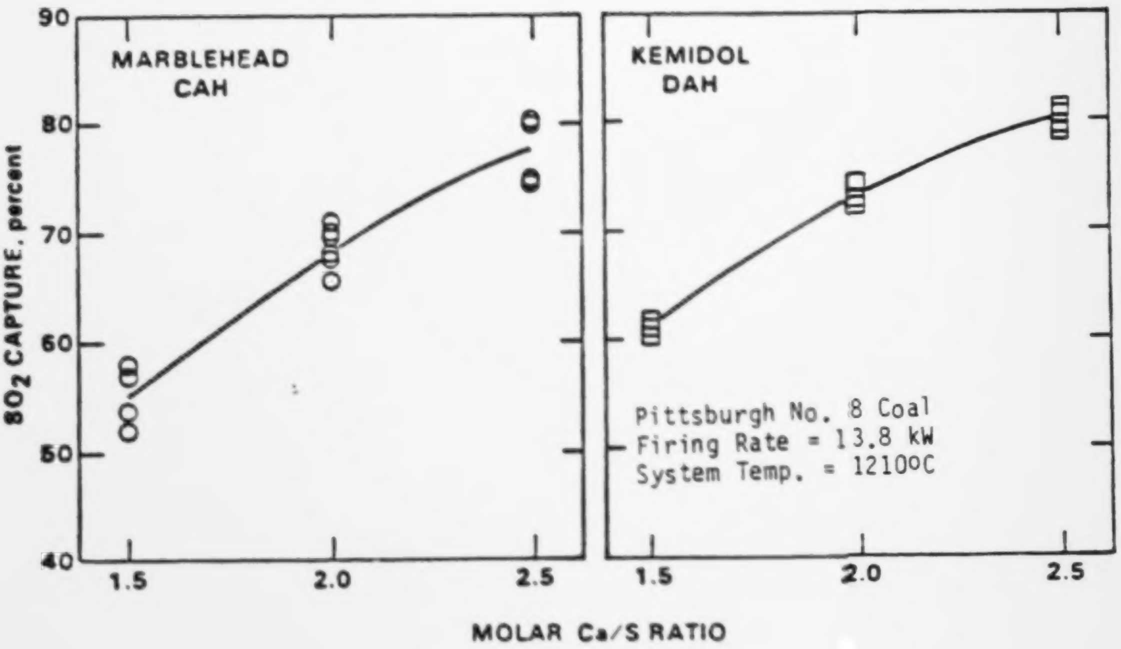


Figure 5-6. Effect of Ca/S Ratio on SO_2 Capture for Marblehead and Kemidol Hydrates

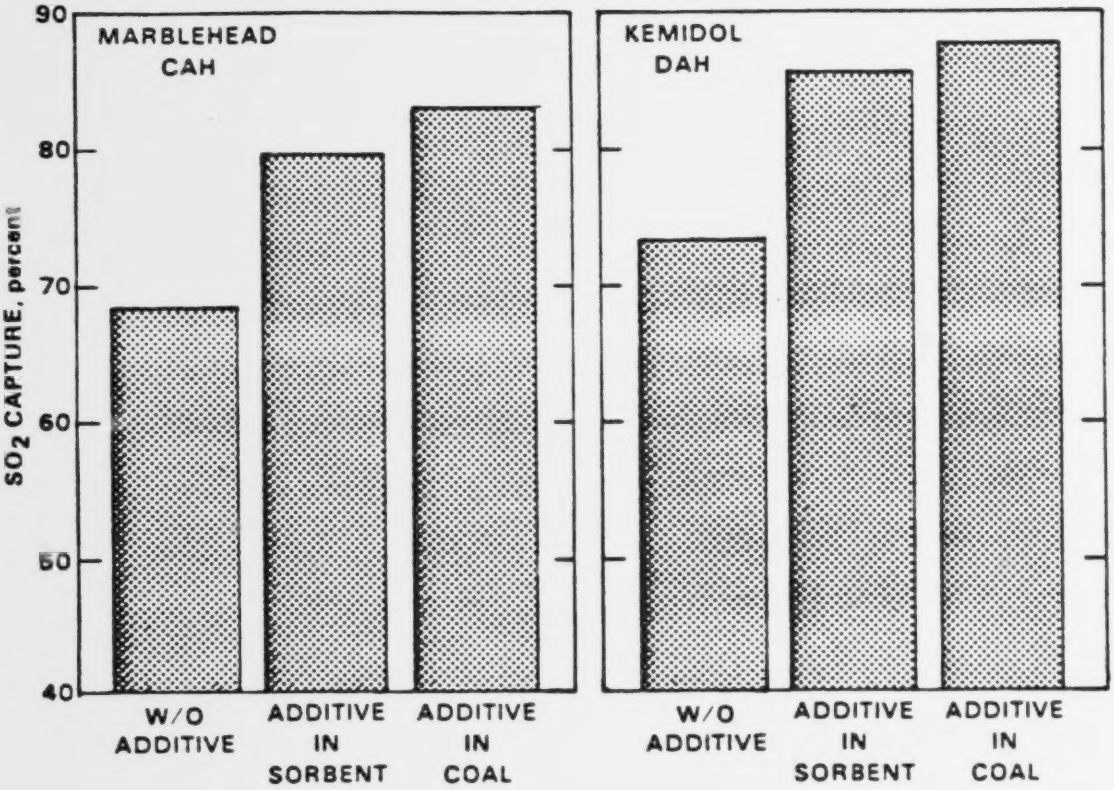


Figure 5-7. Effect of $NaHCO_3$ Additive on SO_2 Capture for Marblehead and Kemidol Hydrates

(Pittsburgh No. 8 Coal, 13.8 kW Firing Rate, 1210°C Injection Temperature) Ca/S = 2, Ca/Na = 15

Compared to the unpromoted measurements, sulfur captures increased by 17% when the additive was mixed with the sorbent and by 20% when mixed with the coal. This behavior may give further insight into the mechanism or promotion. If the promoter contacts the sorbent through vaporization and deposition, then perhaps the higher temperature encountered by the additive in the coal flame enhances this process. In any case, SO_2 removal efficiencies beyond 80% were measured at Ca/S ratios of 2.

The Marblehead and Kemidol sorbents were also tested with the Boich Mine Pittsburgh No. 8 demonstration coal. This was to verify that the Pittsburgh No. 8 baseline coal used throughout the screening tests was providing an accurate representation of sorbent performance with the demonstration fuel. The performance of Marblehead calcitic hydrate remained unchanged at a sulfur removal efficiency of 69%. In contrast, sulfur capture using Kemidol increased substantially from 73% with the baseline coal to 83% for Boich coal. This measurement, however, may be suspect since the dolomitic hydrate proved difficult to feed and time was not available in the schedule for a confirming retest. It is expected, therefore, that all of the sorbents considered in this program will give similar performance levels with both coals.

5.5.5 Conclusions From The Demonstration Sorbent Testing

The results of this test program may be summarized as follows:

1) Testing with Pittsburgh No. 8 coal gave a good representation of the performance of the candidate demonstration sorbents with the Boich mine demonstration coal;

2) At Ca/S molar ratios of 2, maximum measured SO_2 capture efficiencies were 38% for Vicron limestone, 69% for Marblehead calcitic atmospheric

hydrate, and 75% for Ivory Finish pressure hydrated dolomitic lime;

3) Increased SO_2 capture efficiencies occurred at higher Ca/S ratios; however, calcium utilization efficiencies decreased;

4) A 17 to 20% increase in SO_2 capture efficiency resulted when the promoter $NaHCO_3$ was added to the reaction zone at a Ca/Na molar ratio of 15;

5) The promotional effect was maximized when $NaHCO_3$ was mixed with the coal rather than with the sorbent, possibly indicating a process of vaporization and deposition for the additive.

REFERENCES FOR SECTION 5

5.1 Steinberg, M., Yoo, H.J. and McGauley, P.J., "Portland Cement as a Regenerable Sorbent for the Removal and Recovery of SO_2 in the Fluidized Bed Combustion of Coal," U.S. DOE Contract DE-AC02-76CH00016, Brookhaven National Laboratory, Upton, NY, March, 1981.

5.2 Overmoe, B., Chen, S., Ho, L., Seeker, W., Heap, M., and Pershing, D., "Boiler Simulator Studies on Sorbent Utilization for SO_2 Control," In Proceedings: First Joint Symposium on Dry SO_2 and Simultaneous SO_2/NO_x Control Technologies, Volume 1: Fundamental Research and Process Development, EPA-600/9-85-020a (NTIS PB85-232353), July 1985.

5.3 Beittel, R., Gooch, J., Dismukes, E., and Muzio, L., "Studies of Sorbent Calcination and SO_2 Sorbent Reactions in a Pilot Scale Furnace," In Proceedings: First Joint Symposium on Dry SO_2 and Simultaneous SO_2/NO_x Control Technologies, Volume 1: Fundamental Research and Process Development, EPA-600/9-85-020a (NTIS PB85-232353), July 1985.

5.4 Bortz, S. and Flament, P., "Recent IFRF Fundamental and Pilot Scale Studies on the Direct Sorbent Injection Process," In Proceedings: First Joint Symposium on Dry SO_2 and Simultaneous SO_2/NO_x Control Technologies, Volume 1: Fundamental Research and Process Development, EPA-600/9-85-020a (NTIS PB85-232353), July 1985.

SECTION 6

RECOMMENDATIONS

As a result of these sorbent evaluation studies conducted in the EPA Innovative Furnace, a number of recommendations can be made for full scale demonstrations of the technology as well as for future pilot scale studies. Regarding full scale applications of the LIMB technology, it is fairly clear that it is best to use a hydrated sorbent to achieve SO_2 captures of 50% or higher at Ca/S molar ratios of 2. Pressure hydrating appears to give no advantage over atmospheric hydrating. Dolomitic materials will, in general, perform better than calcitics on an equivalent calcium basis (constant Ca/S ratio), but on an equivalent mass basis, their performances appear to be about equal. Thus, the choice between dolomitic or calcitic sorbents will need to be guided by relative availability of the materials at the plant site, capacity of the feed system, and collection efficiency of the particulate emission control devices. Within a given class of sorbent; i.e. carbonate, hydrate, calcitic, or dolomitic, there appears to be little difference in SO_2 capture performance from sorbent to sorbent. Thus, once a class of sorbent has been identified for a particular application, its selection may be based on local availability, market value, and transportation costs, rather than on petrological properties alone. Finally, the use of additives to promote the sulfation reaction may be useful for full scale applications.

For future pilot scale research efforts, further studies should be made to give more complete information on the effect of promotional additives.

Other additive types as well as injection locations and alternate mixing techniques should be evaluated. The combination of boiler sorbent injection with fuel staging or reburning should also be investigated. With reburning, the near-burner environment from a thermal and chemical standpoint may allow sorbent injection lower in the boiler and thus give a greater residence time for SO_2 capture. A potential drawback however, may be the temperature profile through the reburn zone which may deactivate the sorbent. Alternate sorbent injection techniques, such as the injection of a slurry should be investigated. Slurry injection offers a number of potential advantages. A slurry resulting from a slaking process during hydration of a sorbent could be injected directly into the boiler. This would allow the hydrator and the boiler to be closely coupled and would eliminate the need for an intermediate drying step. Limited slurry testing has given some evidence that an inexpensive sorbent such as limestone can achieve relatively high levels of SO_2 capture. The use of alternative sorbents such as alkali based materials or mixed cations as suggested by Ham et al. (6-1) should be investigated at the pilot scale. Also better overall sorbent utilization may result by recycling through the boiler. Pilot scale testing could address the many questions remaining regarding the reactivity of a mix of ash and partially spent sorbent. Finally, pilot scale studies of boiler sorbent injection combined with downstream or coolside capture should be undertaken. One scheme which may allow the use of inexpensive sorbents is the injection of limestone in the boiler to calcine and give partial sulfation. After passing through the convective heat transfer section, the flue gas and partially sulfated sorbent could could be further activated by humidification. Sulfation would continue until the sorbent and flyash is removed in the particulate emission control device. Pilot scale process development of this integrated SO_2 control strategy is required for ultimate full scale success.

REFERENCES FOR SECTION 6

6.1 Ham, D.O., Simons, G.A. and Moniz, G.A., "Alternative SO_2 Sorbents", In Proceedings: 1986 Joint Symposium on Dry SO_2 and Simultaneous SO_2/NO_x Control Technologies, Volume I: Sorbents Process Research, and Dispersion, EPA-600/9-86-029a, October, 1986.

APPENDIX A

FACILITY DRAWINGS

Page

A.1 Furnace Sections A-2

A.2 Coal and sorbent Feeder Systems A-24

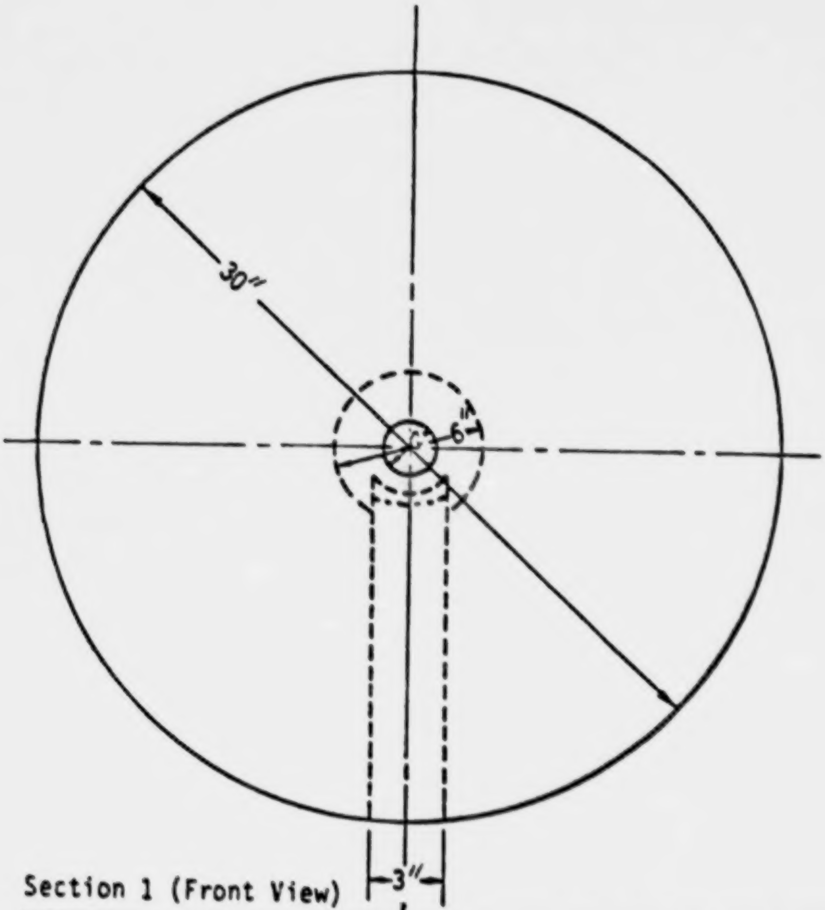
A.3 Sorbent Injection and Sample Probes A-30

A.4 Air and Fuel Feed Controls and Electrical Schematics A-43

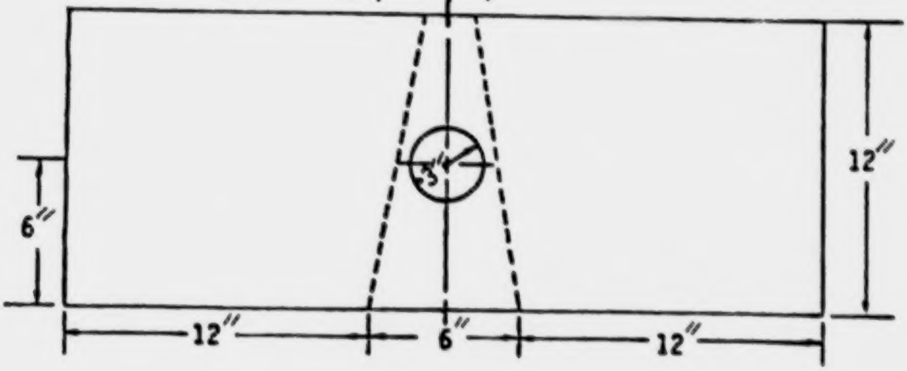
APPENDIX A.1

FURNACE SECTIONS

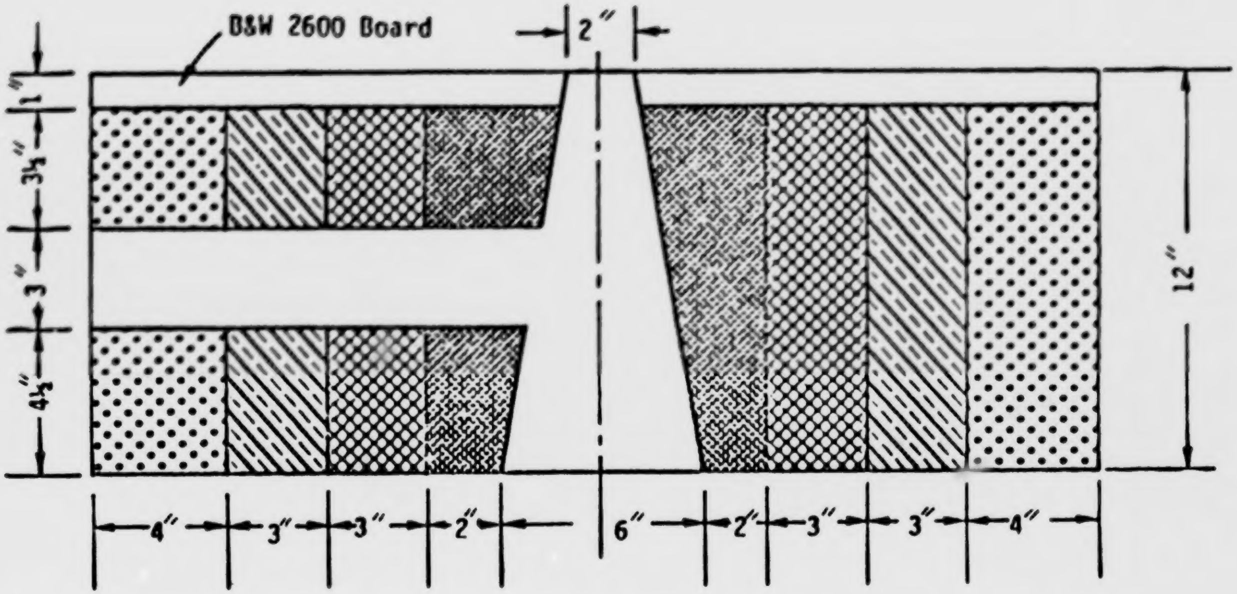
Section 1 (Top View)



Section 1 (Front View)



Section 1 (Side View)


B&W 2600 Board
2"
1"
3½"
3"
4½"
12"
4"
3"
3"
2"
6"
2"
3"
3"
4"

Refractory Legend

▩ Greencast 97

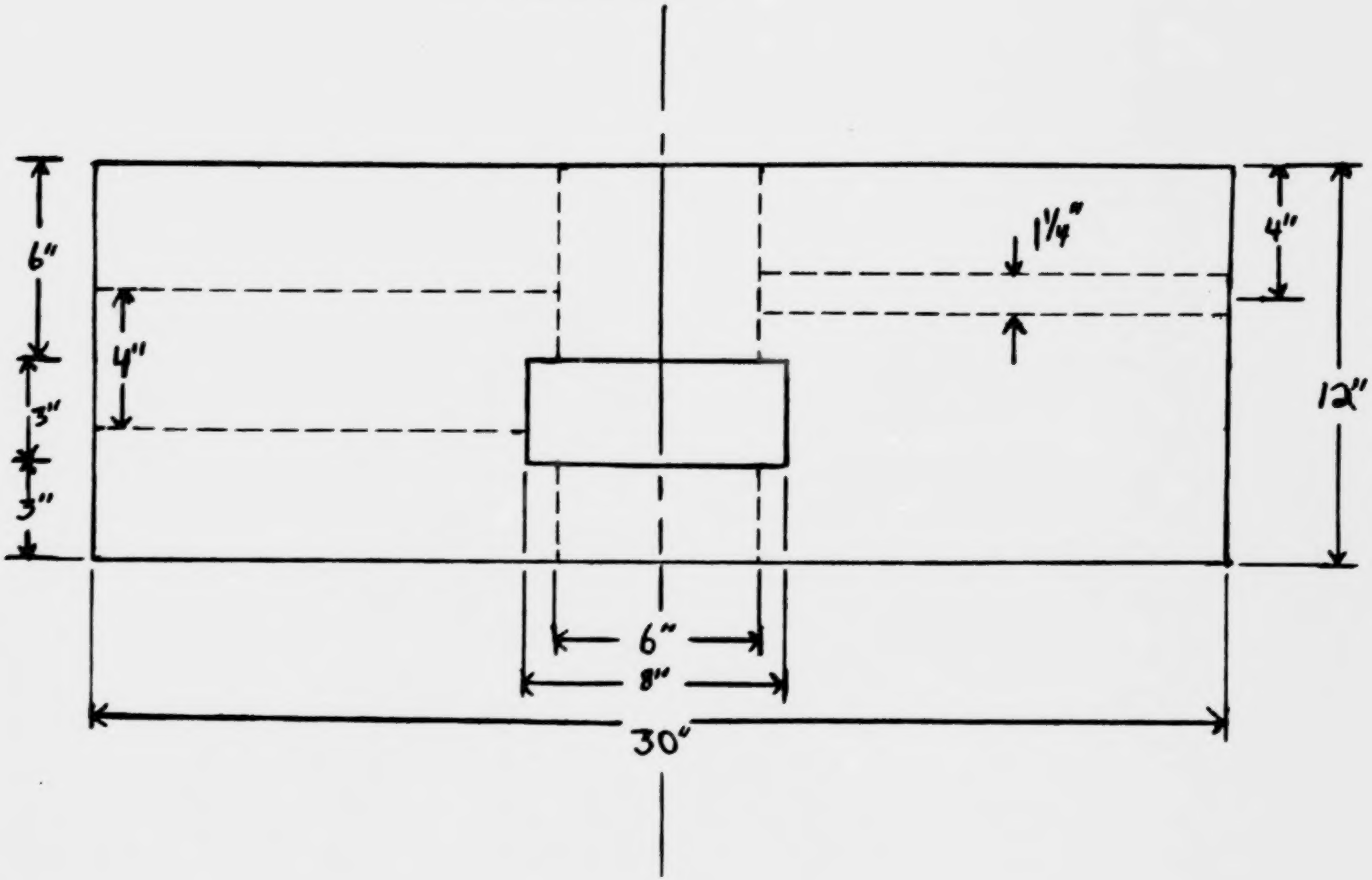
▦ Kast-O-Lite 30

▧ Castable Insulation #22

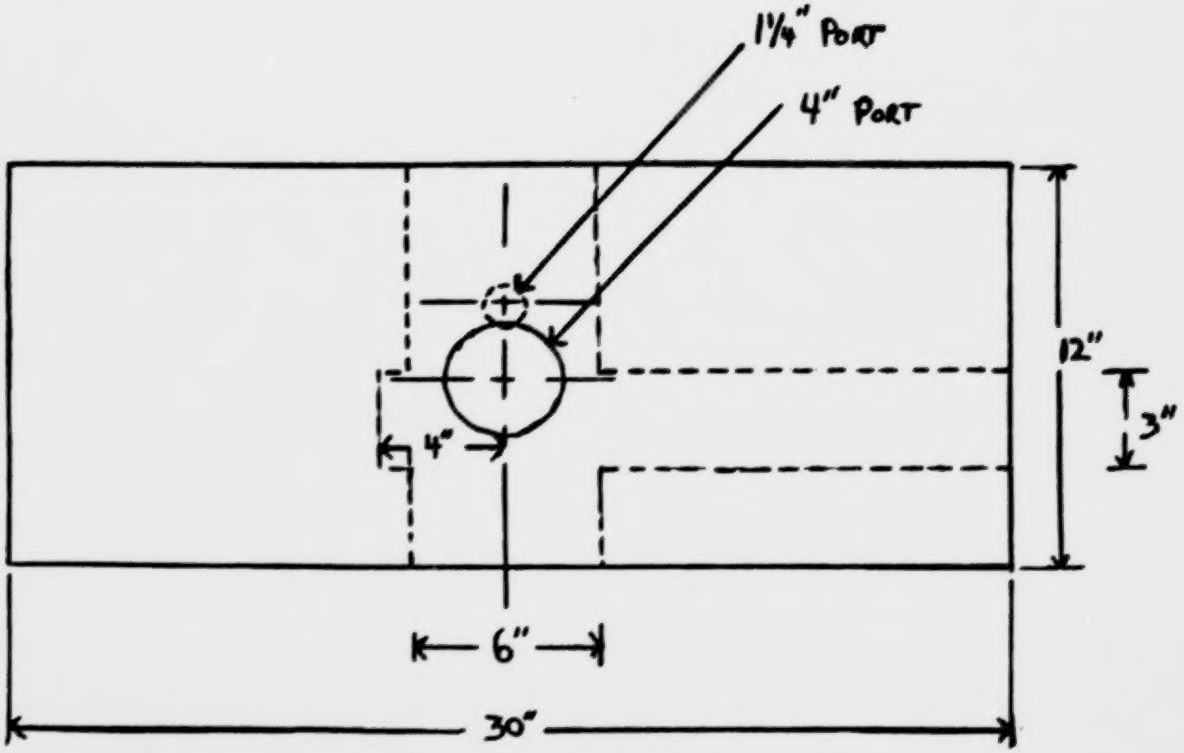
░ Castable Block Mix

FURNACE SECTION # 2

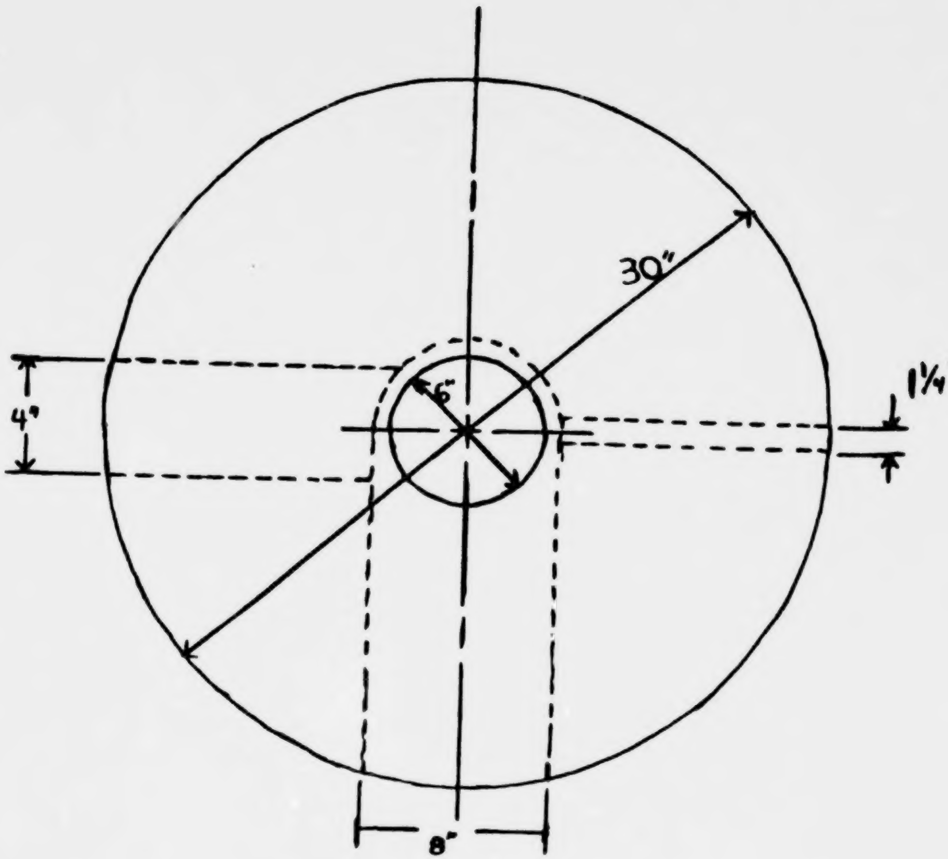
FRONT VIEW


6"
4"
3"
3"
1¼"
4"
12"
6"
8"
30"

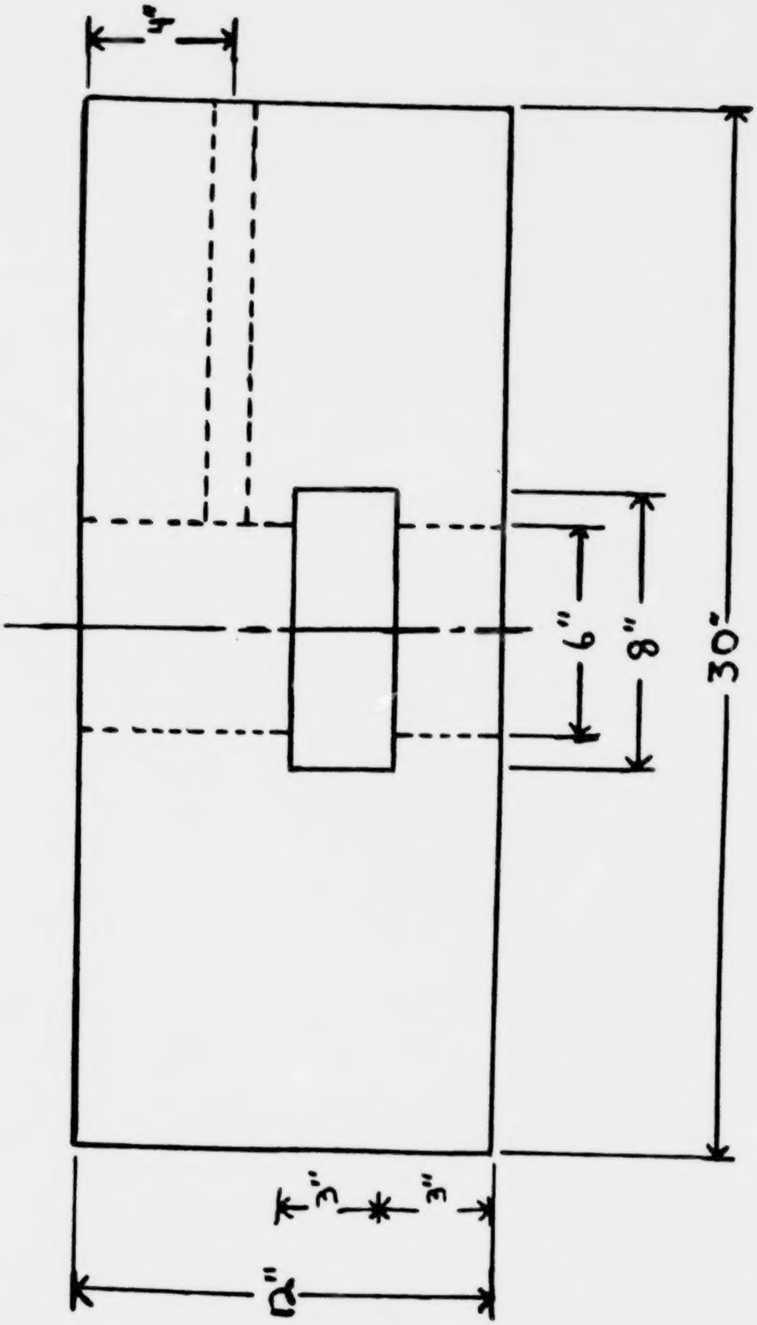
FURNACE SECTION # 2
LEFT SIDE VIEW


1¼" PORT
4" PORT
12"
3"
4"
6"
30"

FURNACE SECTION #2
TOP VIEW

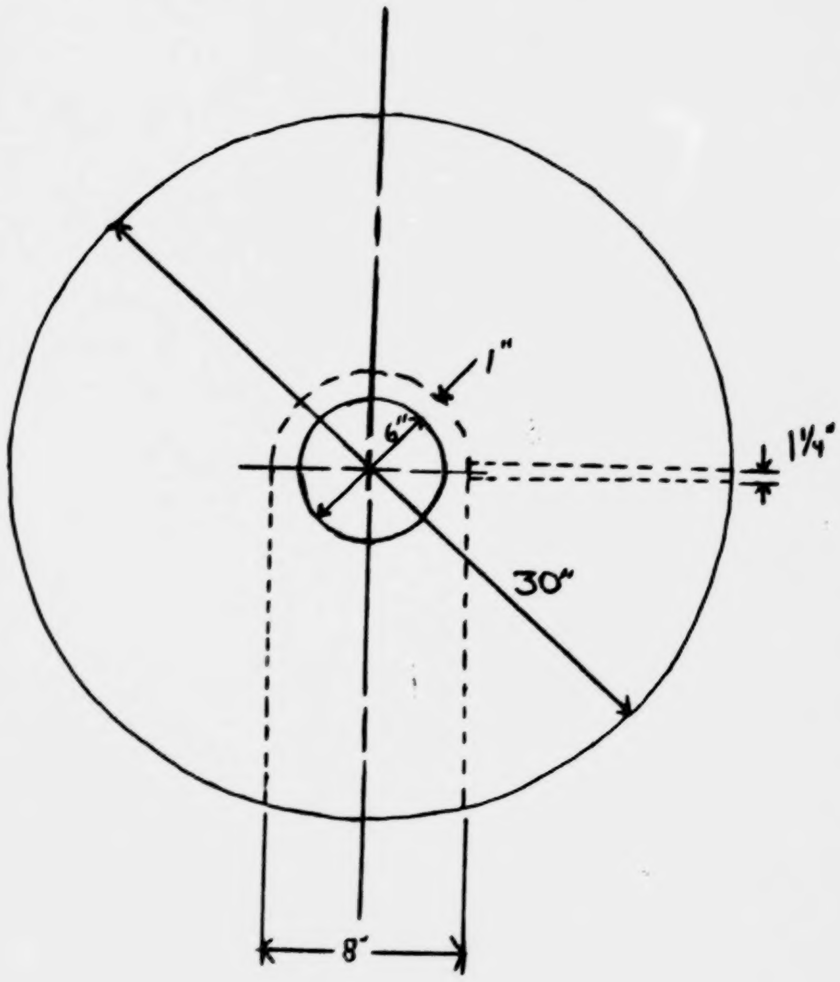


FURNACE SECTION #3
FRONT VIEW

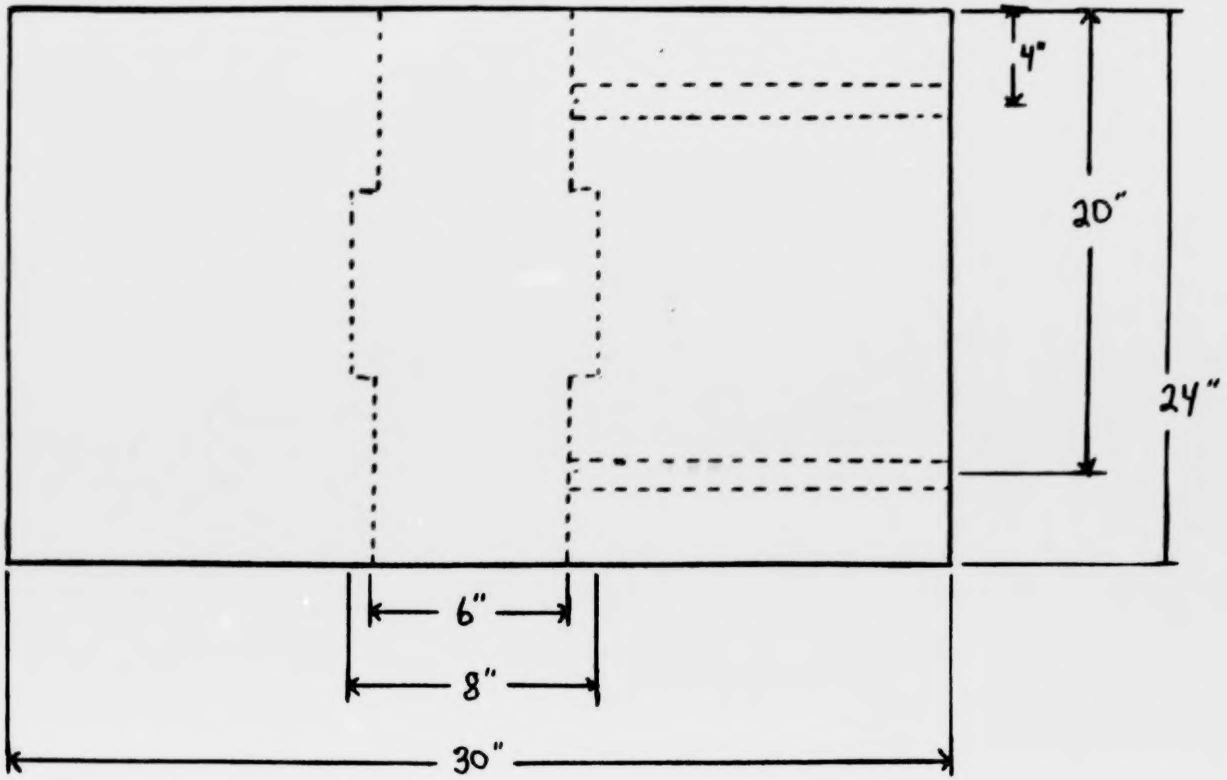


FURNACE SECTIONS # 3, 4, 5, 6

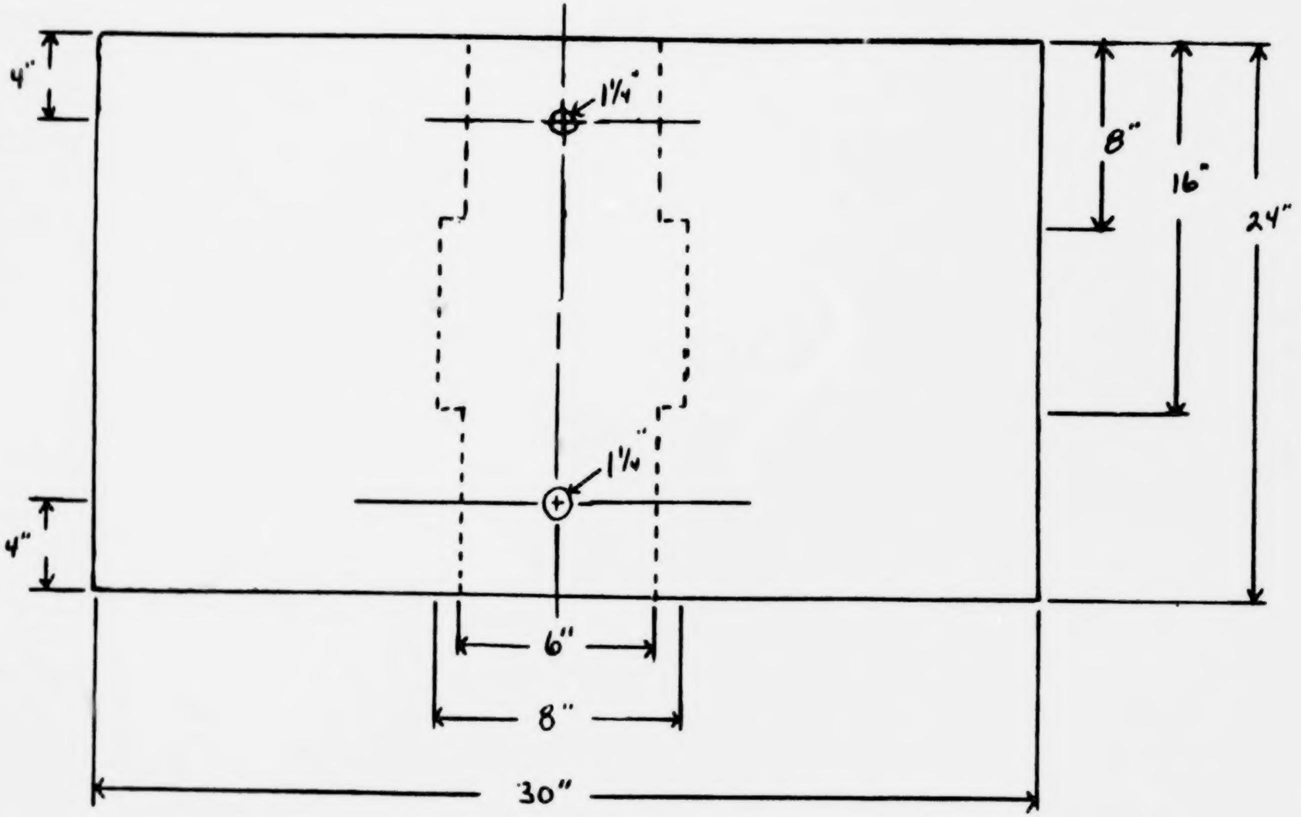
TOP VIEW



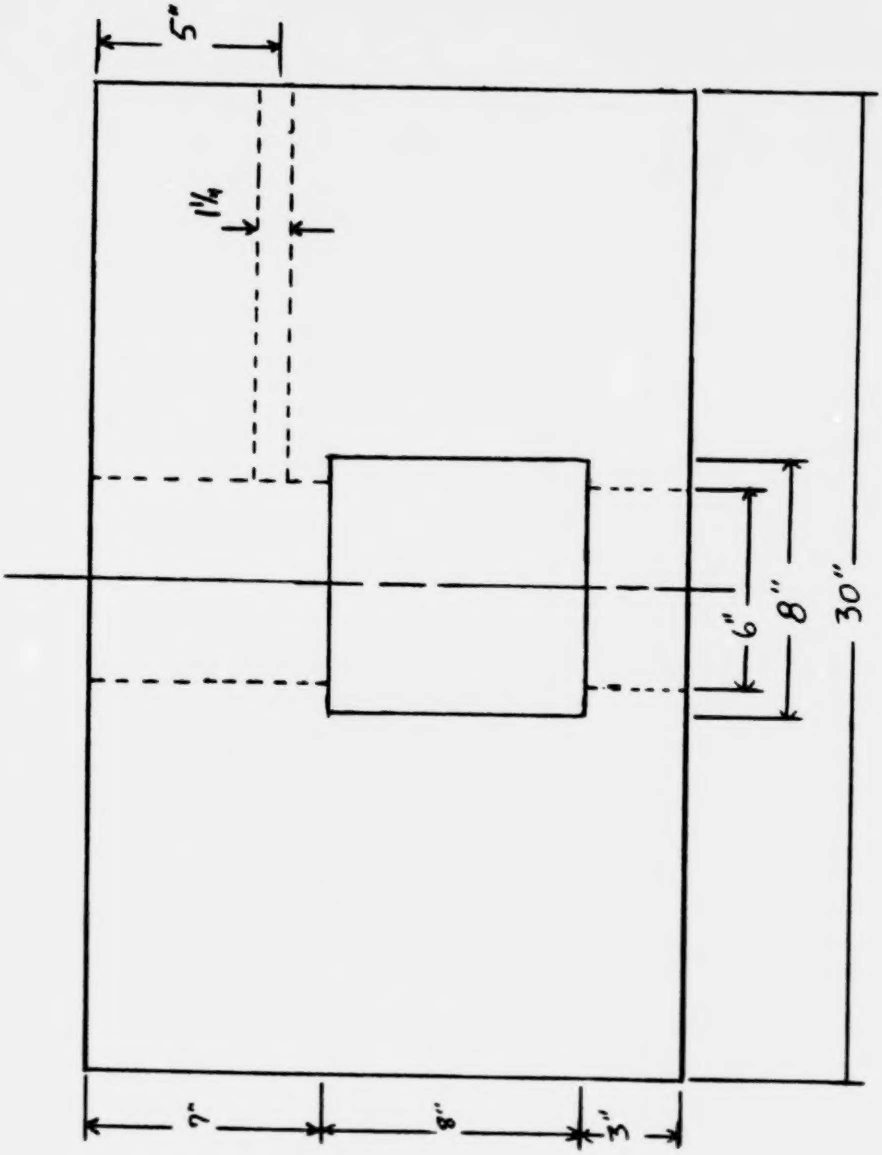
FURNACE SECTION # 4
Front View



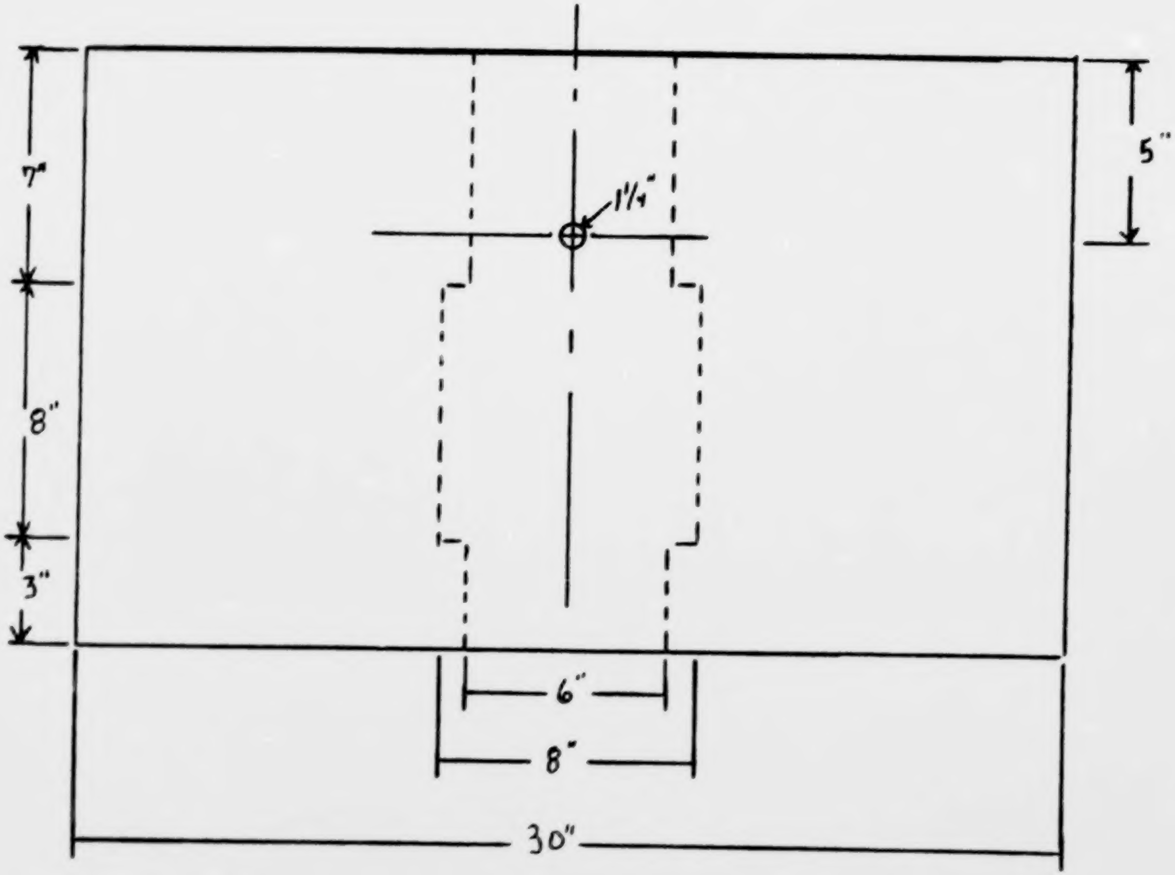
FURNACE SECTION # 4
SIDE VIEW


1¼"
1¼"
4"
4"
8"
16"
24"
6"
8"
30"

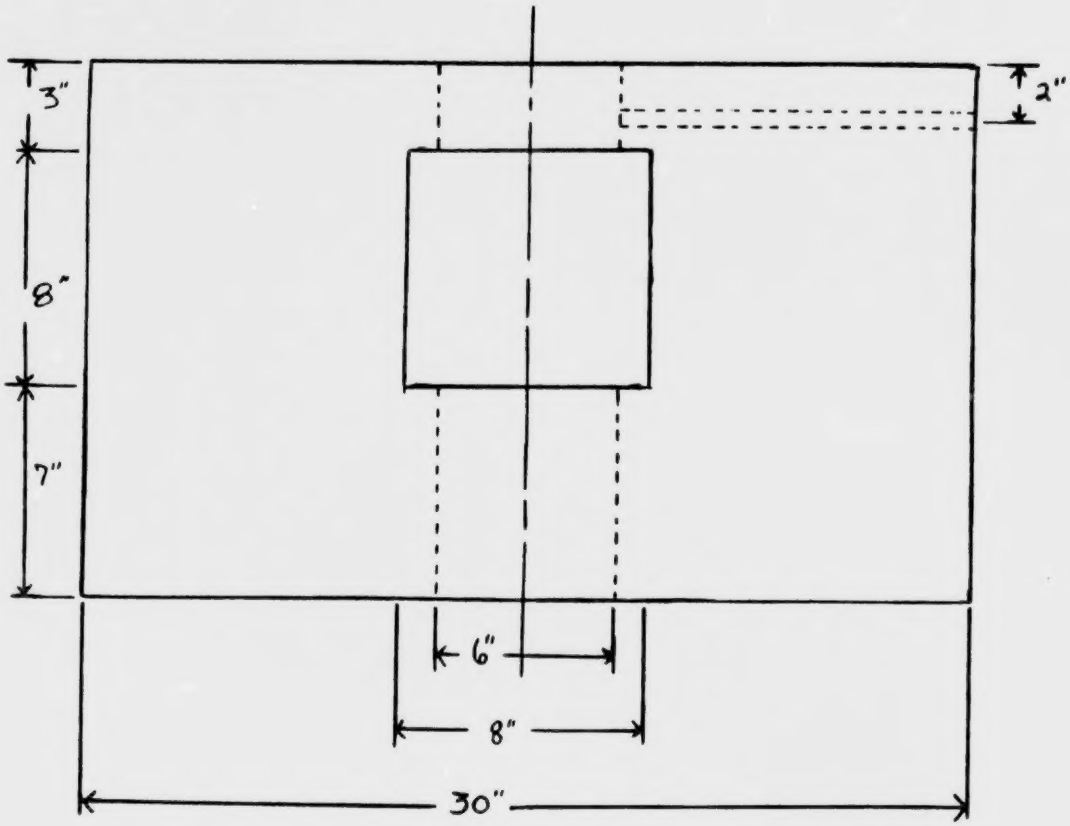
FURNACE SECTION #5
FRONT VIEW


5"
1¼
6"
8"
30"
7"
8"
3"

FURNACE SECTION #5
SIDE VIEW


7"
8"
3"
1¼"
5"
6"
8"
30"

FURNACE SECTION # 6
FRONT VIEW



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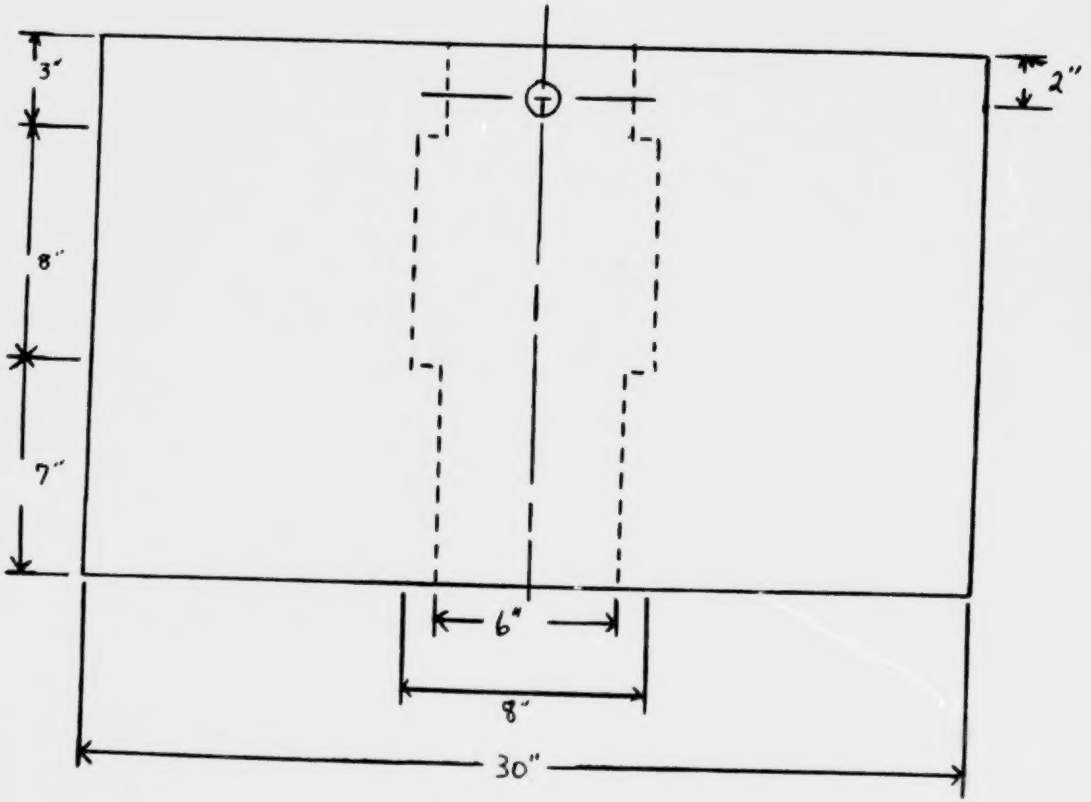
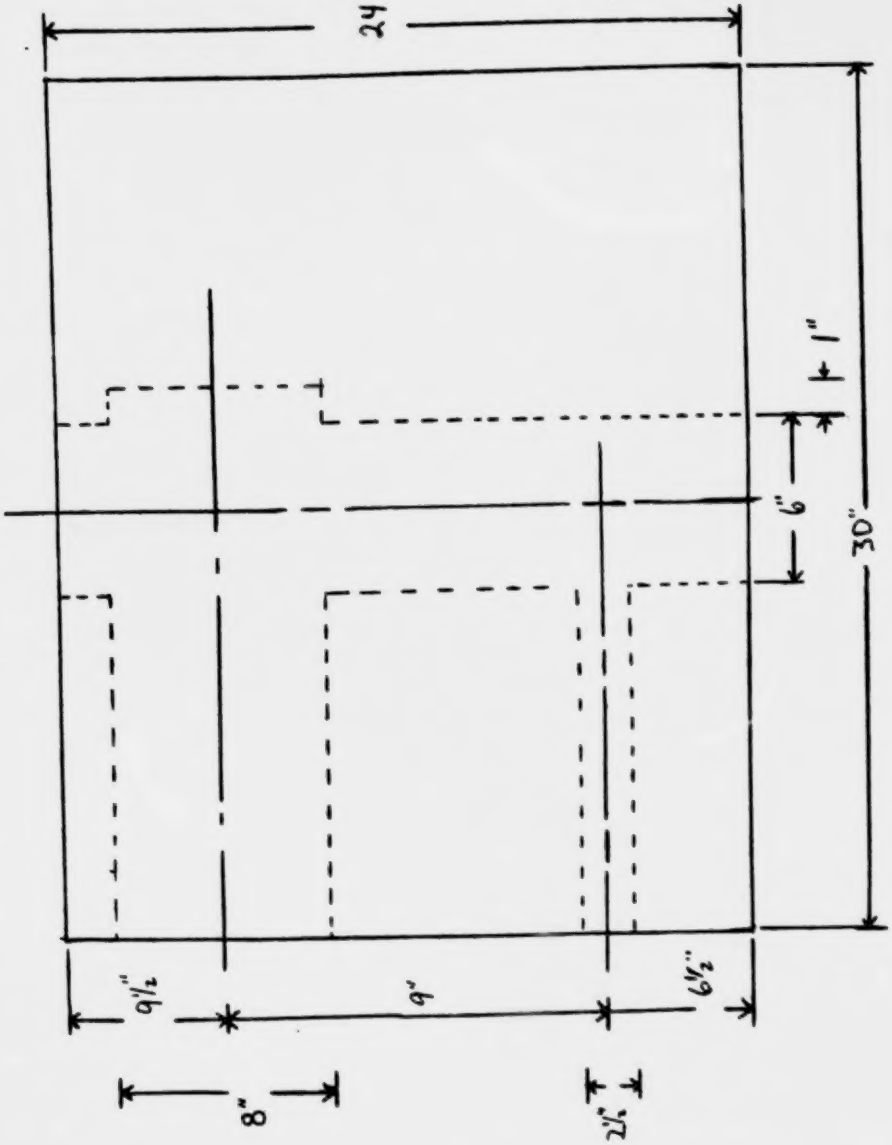
SIDE VIEW

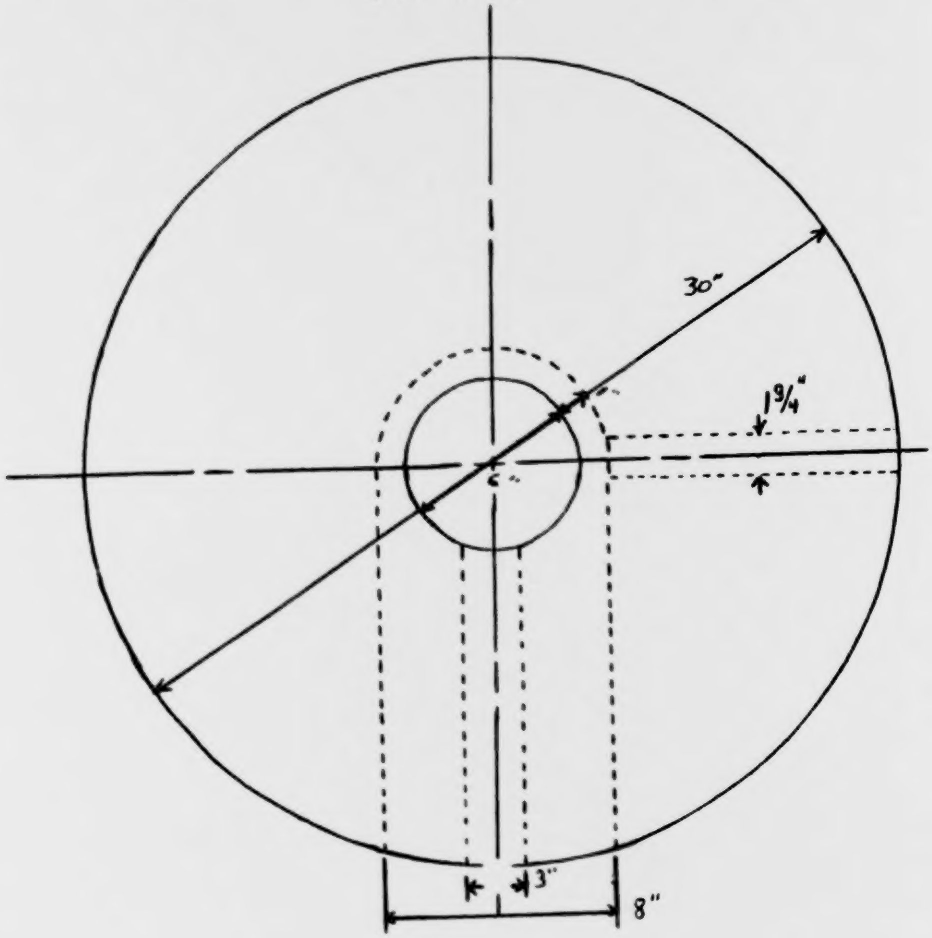




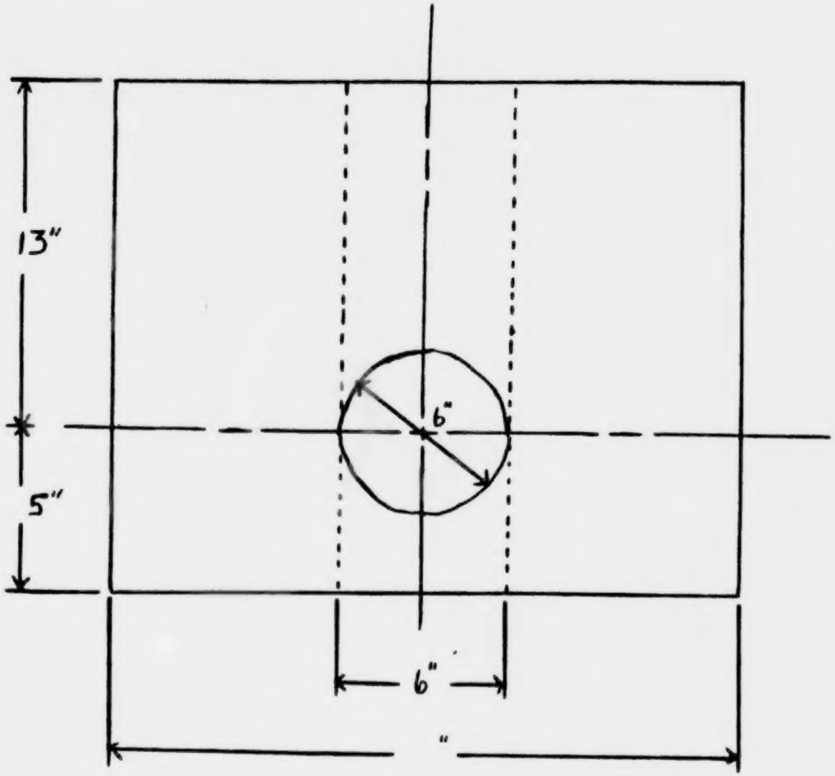
FURNACE SECTION # 7
SIDE VIEW

24"
30"
6"
1"
9½"
9"
6½"
8"
2½"

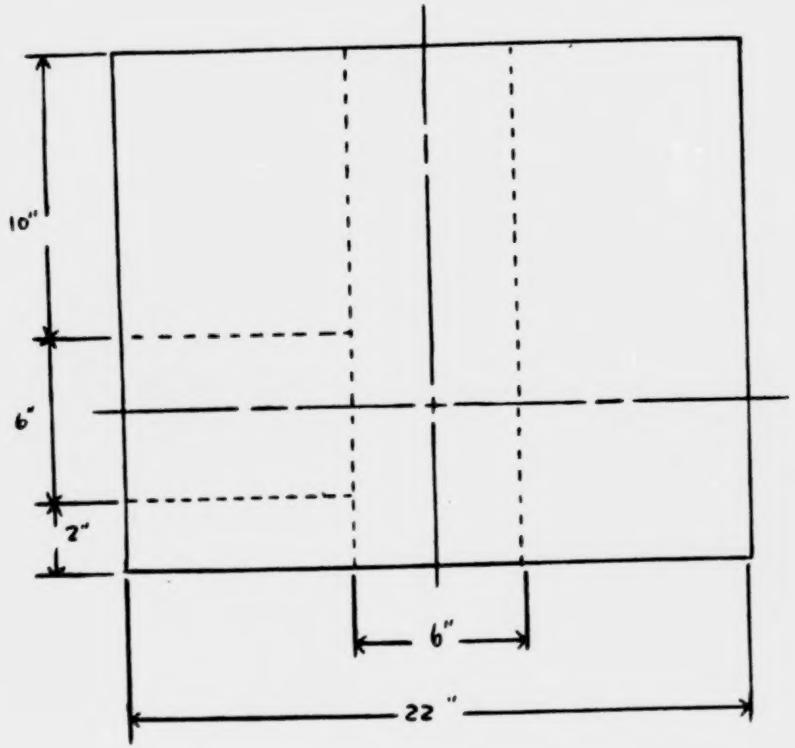
FURNACE SECTION # 7
TOP VIEW

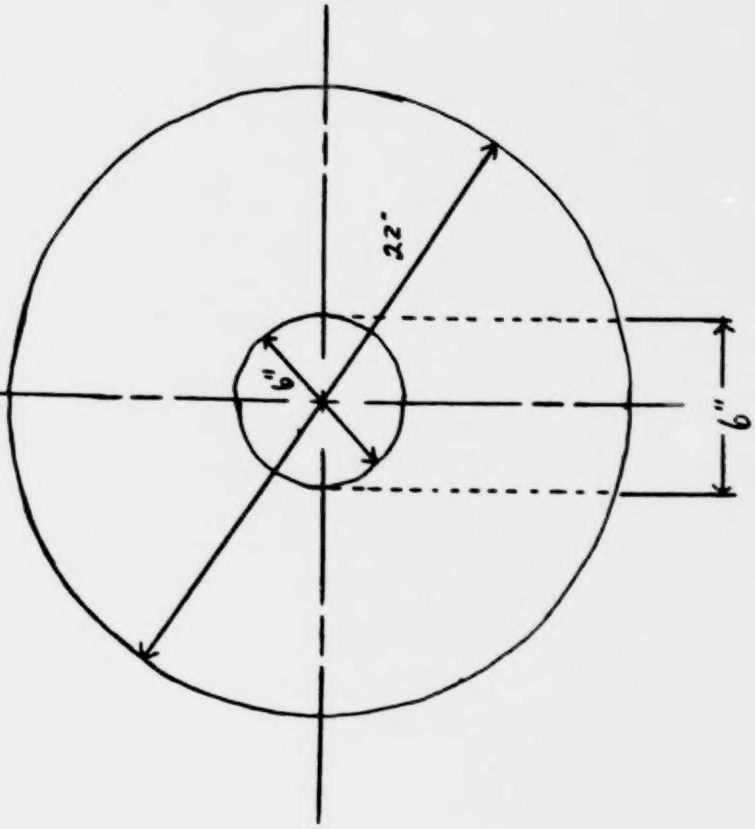


FURNACE SECTION # 8
FRONT VIEW



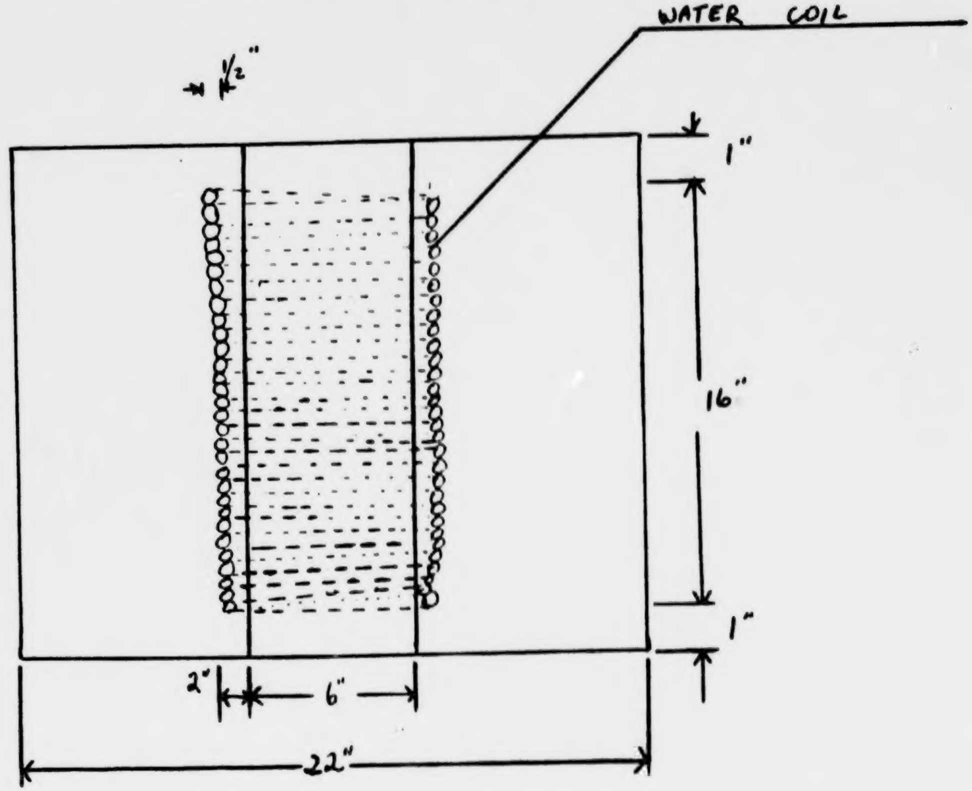
FURNACE SECTION # 8
SIDE VIEW



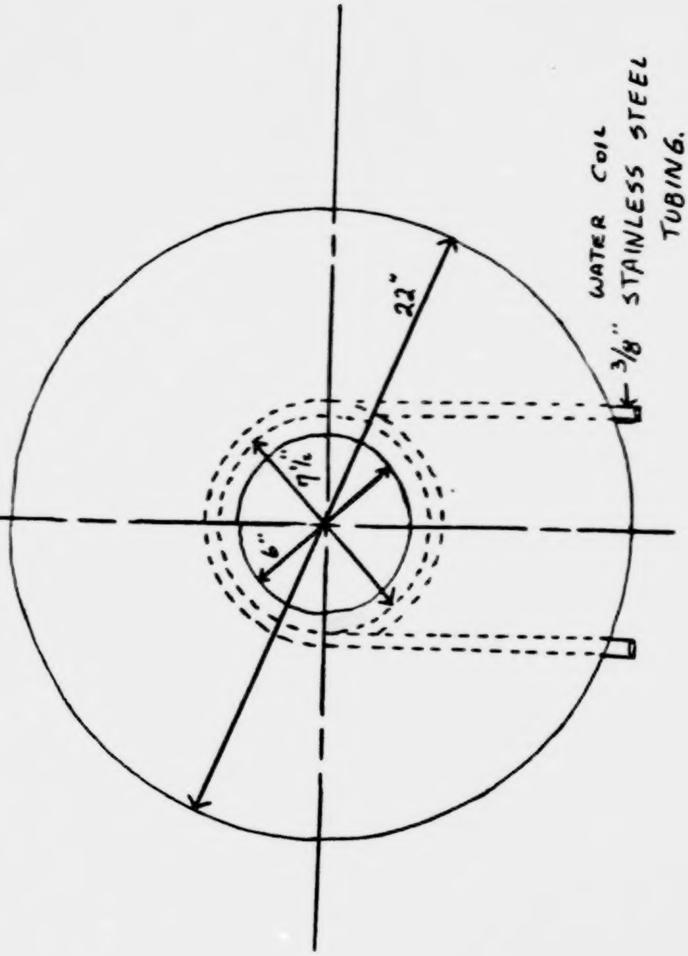


FURNACE SECTION # 8
TOP VIEW

FURNACE SECTION #9 (WATER JACKETED)
FRONT CUT AWAY VIEW

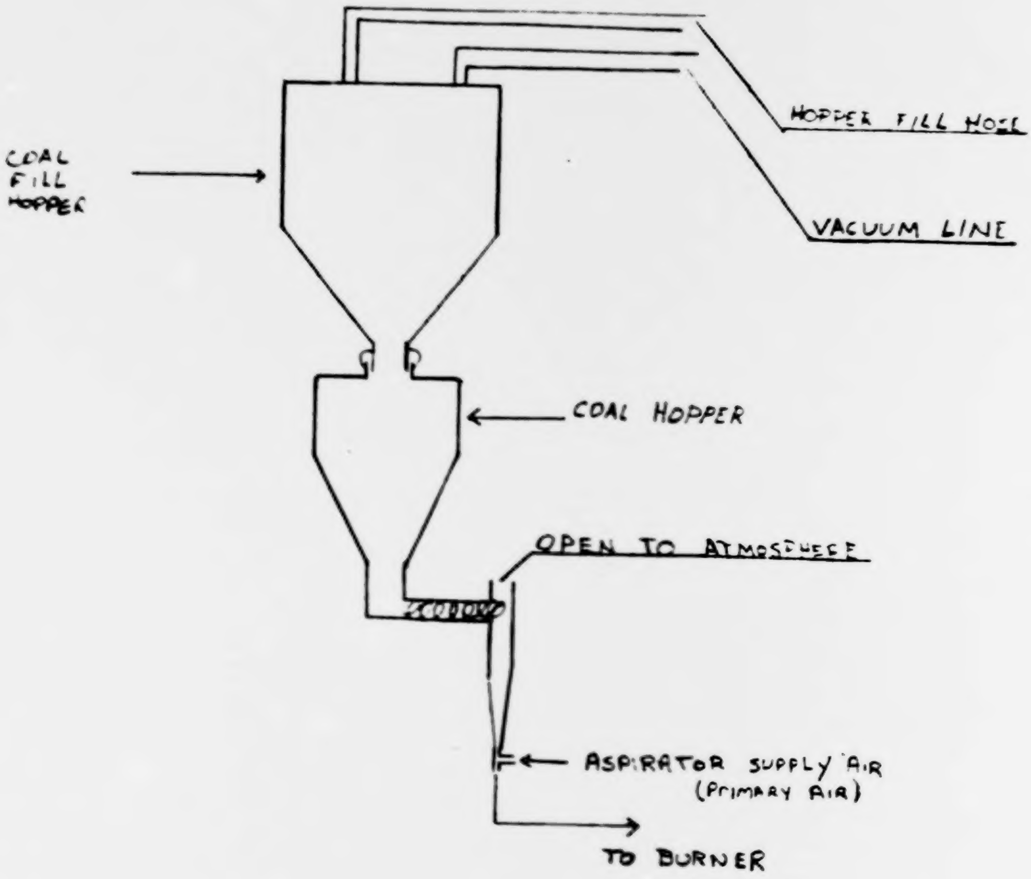

WATER COIL
1/2"
1"
16"
1"
2"
6"
22"

FURNACE SECTION # 9
TOP VIEW

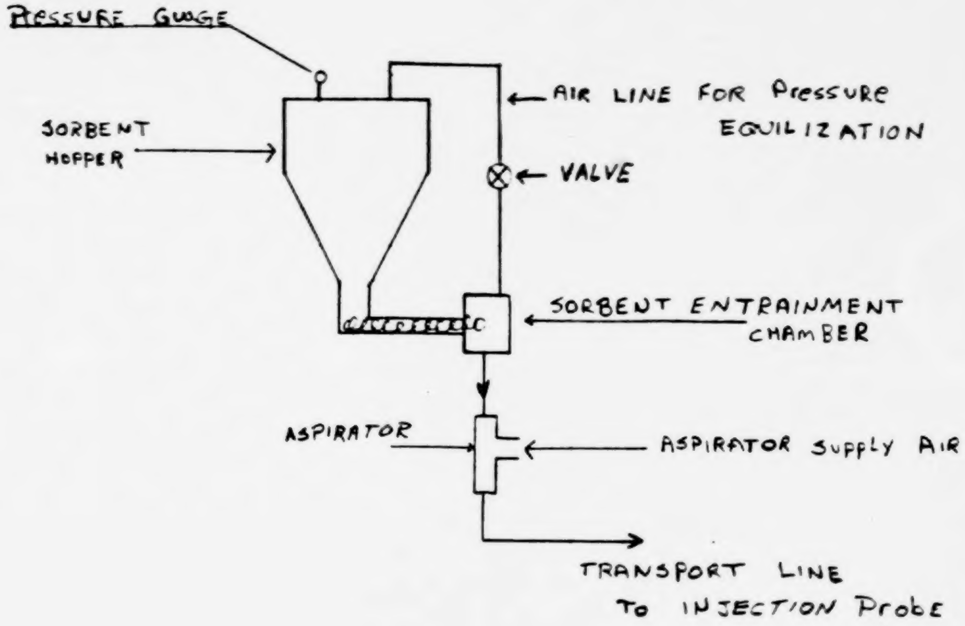

22"
7½"
6"
WATER COIL
3/8" STAINLESS STEEL
TUBING.

APPENDIX A.2

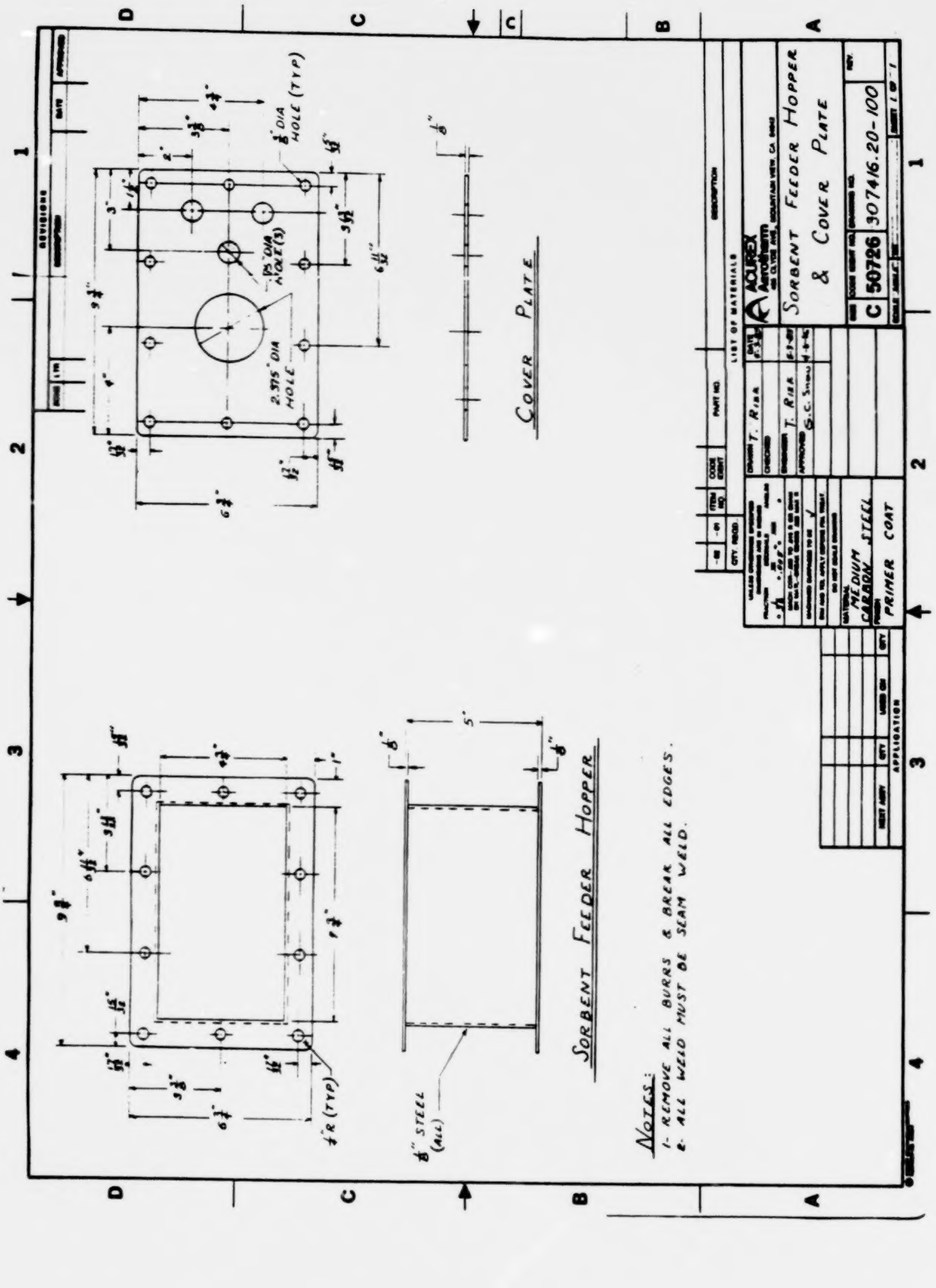
COAL AND SORBENT FEEDER SYSTEMS

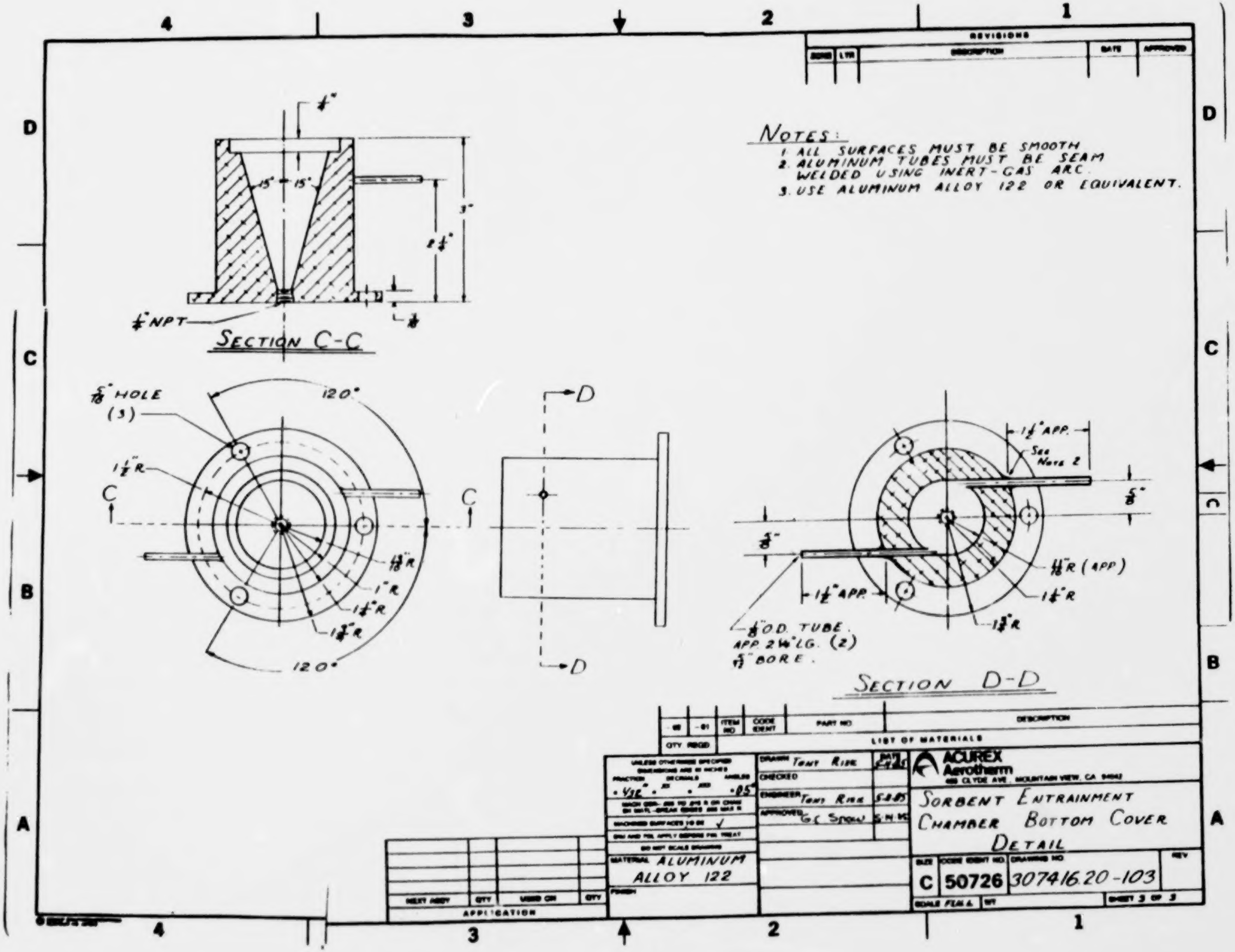

COAL FILL HOPPER
HOPPER FILL HOSE
VACUUM LINE
COAL HOPPER
OPEN TO ATMOSPHERE
ASPIRATOR SUPPLY AIR (PRIMARY AIR)
TO BURNER

COAL FEEDER SCHEMATIC


PRESSURE GUAGE
AIR LINE FOR PRESSURE EQUILIZATION
SORBENT HOPPER
VALVE
SORBENT ENTRAINMENT CHAMBER
ASPIRATOR
ASPIRATOR SUPPLY AIR
TRANSPORT LINE TO INJECTION Probe

SORBENT FEEDER Schematic


SORBENT FEEDER HOPPER
COVER PLATE
2.375" DIA HOLE
.75" DIA HOLE (3)
1/8" DIA HOLE (TYP)
1/4" R (TYP)
1/8" STEEL (ALL)
NOTES:
1- REMOVE ALL BURRS & BREAK ALL EDGES.
2- ALL WELD MUST BE SEAM WELD.
REVISIONS
LIST OF MATERIALS
ACUREX Aerotherm
MOUNTAIN VIEW, CA
DRAWN T. RIBA
ENGINEER T. RIBA
APPROVED G.C. SNOW
MATERIAL MEDIUM CARBON STEEL
FINISH PRIMER COAT
SORBENT FEEDER HOPPER & COVER PLATE
C 50726 307416.20-100
SHEET 1 OF 1
APPLICATION


SECTION C-C
1/4"
15°
15°
3"
2 1/4"
1/8
1/4" NPT
5/16" HOLE (3)
120°
120°
1 1/2"R
C
C
D
D
13/16"R
1"R
1 1/4"R
1 3/8"R
1 1/2" APP.
See Note 2
5/8"
5/8"
11/16"R (APP)
1 1/4"R
1 3/8"R
1 1/2" APP.
1/8" O.D. TUBE. APP. 2 1/4" LG. (2) 1/16" BORE.
SECTION D-D
REVISIONS
DESCRIPTION
DATE
APPROVED
LIST OF MATERIALS
PART NO
DESCRIPTION
QTY REQD
APPLICATION
1
2
3
4
A
B
C
D

NOTES:
1. ALL SURFACES MUST BE SMOOTH
2. ALUMINUM TUBES MUST BE SEAM WELDED USING INERT-GAS ARC.
3. USE ALUMINUM ALLOY 122 OR EQUIVALENT.

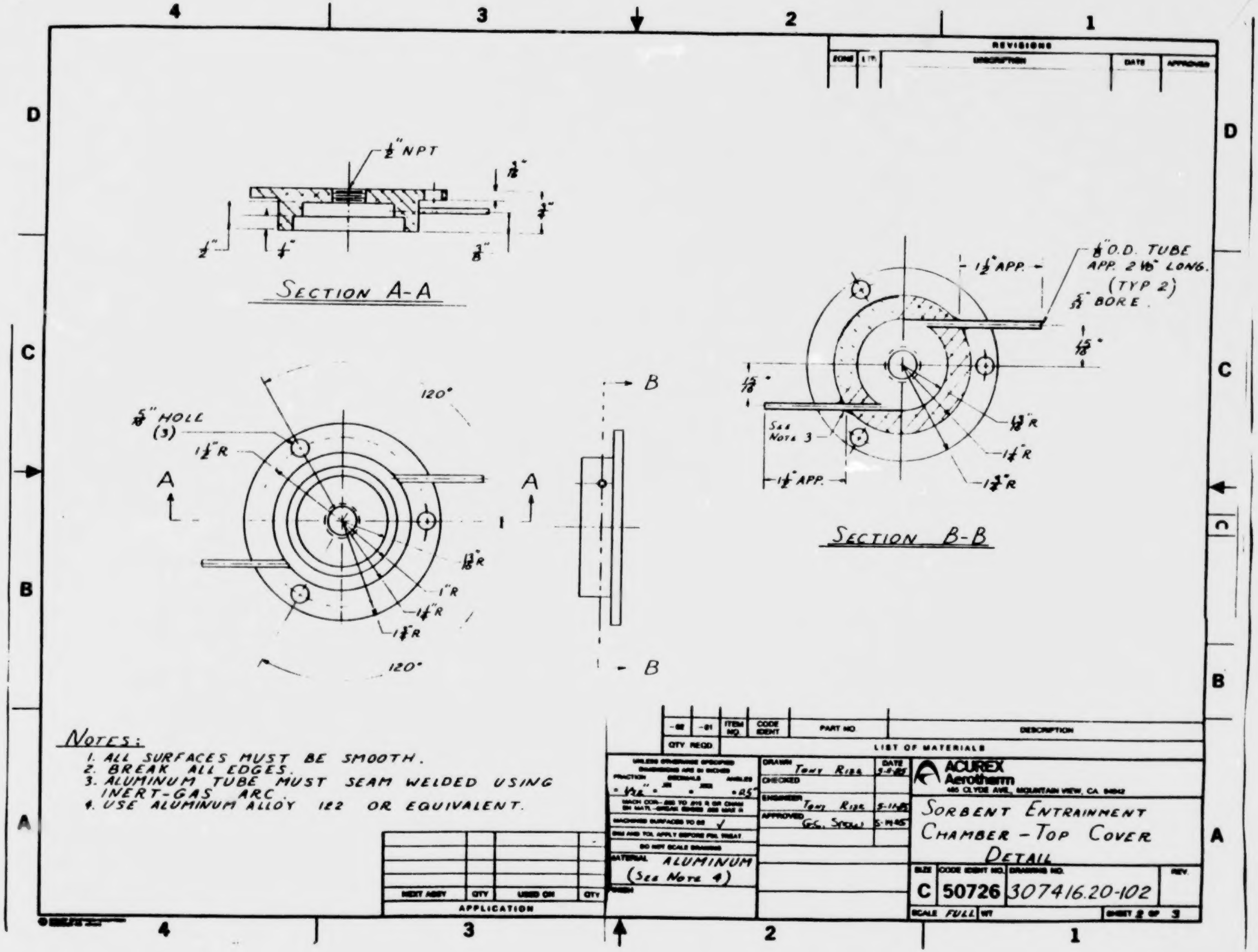
DRAWN Tony Rise
ENGINEER Tony Rise 5-2-85
APPROVED G.C. Snow

MATERIAL ALUMINUM ALLOY 122

ACUREX Aerotherm
485 CLYDE AVE., MOUNTAIN VIEW, CA 94042

SORBENT ENTRAINMENT CHAMBER BOTTOM COVER DETAIL

SIZE C
CODE IDENT NO. 50726
DRAWING NO. 307416.20-103
SHEET 3 OF 3



REVISIONS
ZONE
LTR
DESCRIPTION
DATE
APPROVED
1/2" NPT
1/16"
3/4"
1/2"
1/4"
3/8"
SECTION A-A
5/16" HOLE (3)
1 1/2" R
A
A
120°
120°
13/16" R
1" R
1 1/4" R
1 3/8" R
B
B
1/8" O.D. TUBE APP. 2 1/16" LONG. (TYP 2)
5/32" BORE.
1 1/2" APP.
15/16"
15/16"
See Note 3
1 1/2" APP.
13/16" R
1 1/4" R
1 3/8" R
SECTION B-B
NEXT ASSY
QTY
USED ON
QTY
APPLICATION
1
2
3
4
A
B
C
D

NOTES:

1. ALL SURFACES MUST BE SMOOTH.
2. BREAK ALL EDGES.
3. ALUMINUM TUBE MUST SEAM WELDED USING INERT-GAS ARC.
4. USE ALUMINUM ALLOY 122 OR EQUIVALENT.

-02	-01	ITEM NO	CODE IDENT	PART NO	DESCRIPTION
QTY REQD					

LIST OF MATERIALS

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES
FRACTION ± 1/32" DECIMALS .XX ± .XXX ± ANGLES ± .25°
MACHINED SURFACES TO BE ✓
DO NOT SCALE DRAWING
MATERIAL ALUMINUM (See Note 4)

DRAWN Tony Rise DATE 5-8-85
CHECKED
ENGINEER Tony Rise 5-14-85
APPROVED G.C. Snow 5-14-85

ACUREX Aerotherm
485 CLYDE AVE., MOUNTAIN VIEW, CA 94042

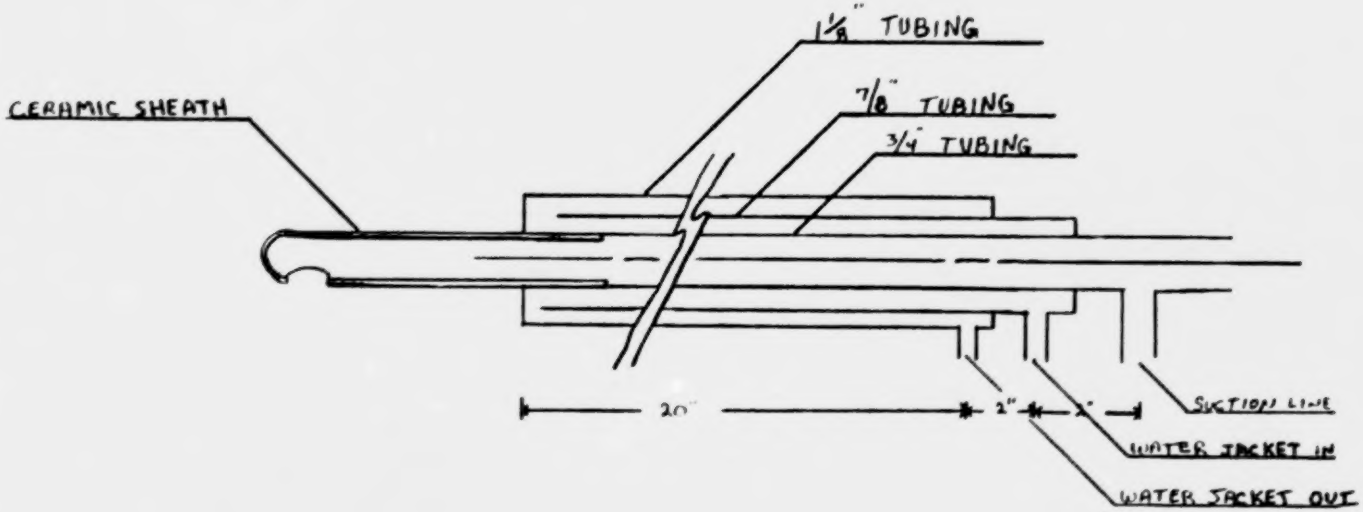
SORBENT ENTRAINMENT CHAMBER - TOP COVER DETAIL

SIZE C CODE IDENT NO. 50726 DRAWING NO. 307416.20-102 REV.
SCALE FULL WT SHEET 2 OF 3

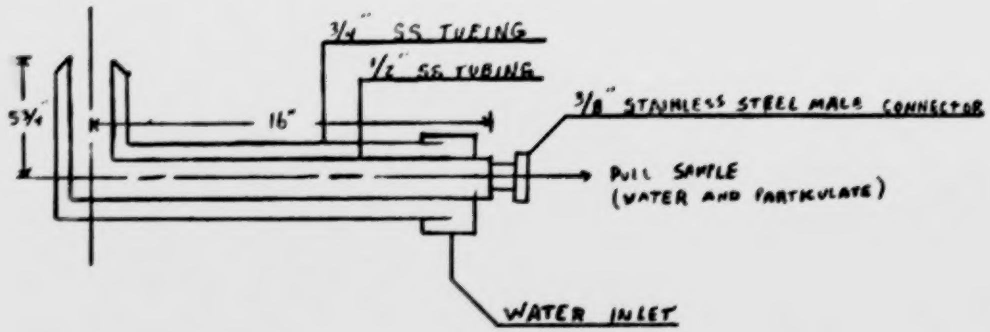
APPENDIX A.3

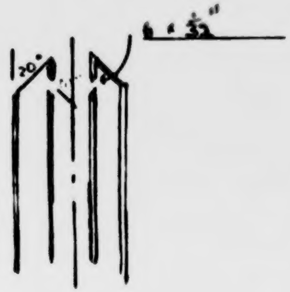
SORBENT INJECTION AND SAMPLE PROBES

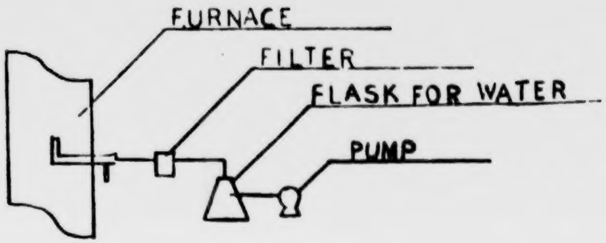
SUCTION PYROMETER


CERAMIC SHEATH
1⅛" TUBING
7/8" TUBING
3/4" TUBING
SUCTION LINE
WATER JACKET IN
WATER JACKET OUT
20"
2"
2"

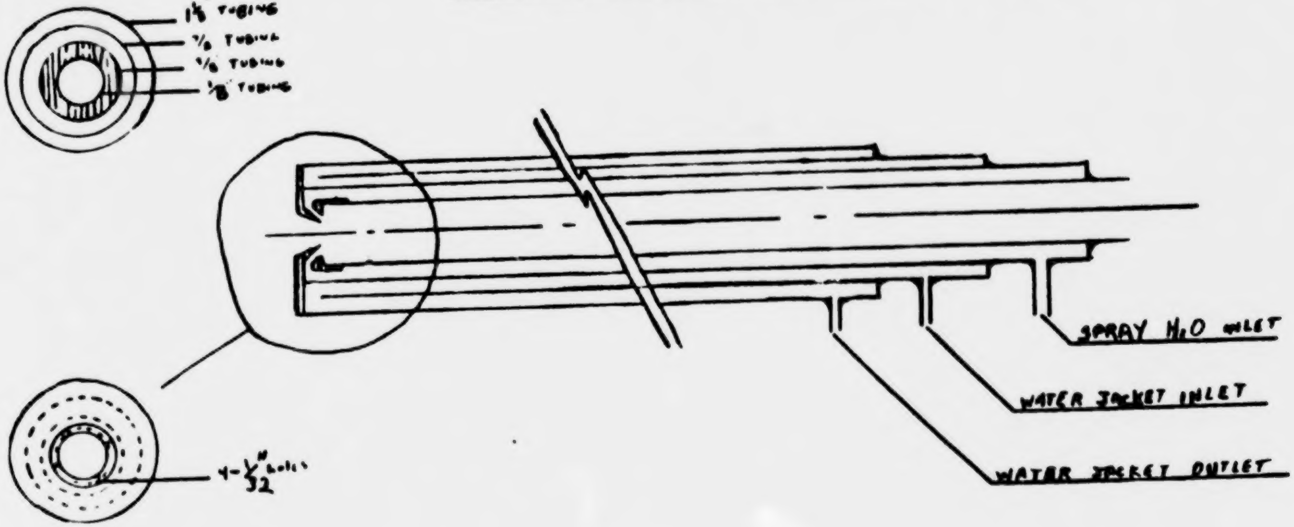
PARTICULATE SAMPLING PROBE


3/4" SS TUBING
1/2" SS TUBING
3/8" STAINLESS STEEL MALE CONNECTOR
5 3/4
16"
PULL SAMPLE (WATER AND PARTICULATE)
WATER INLET



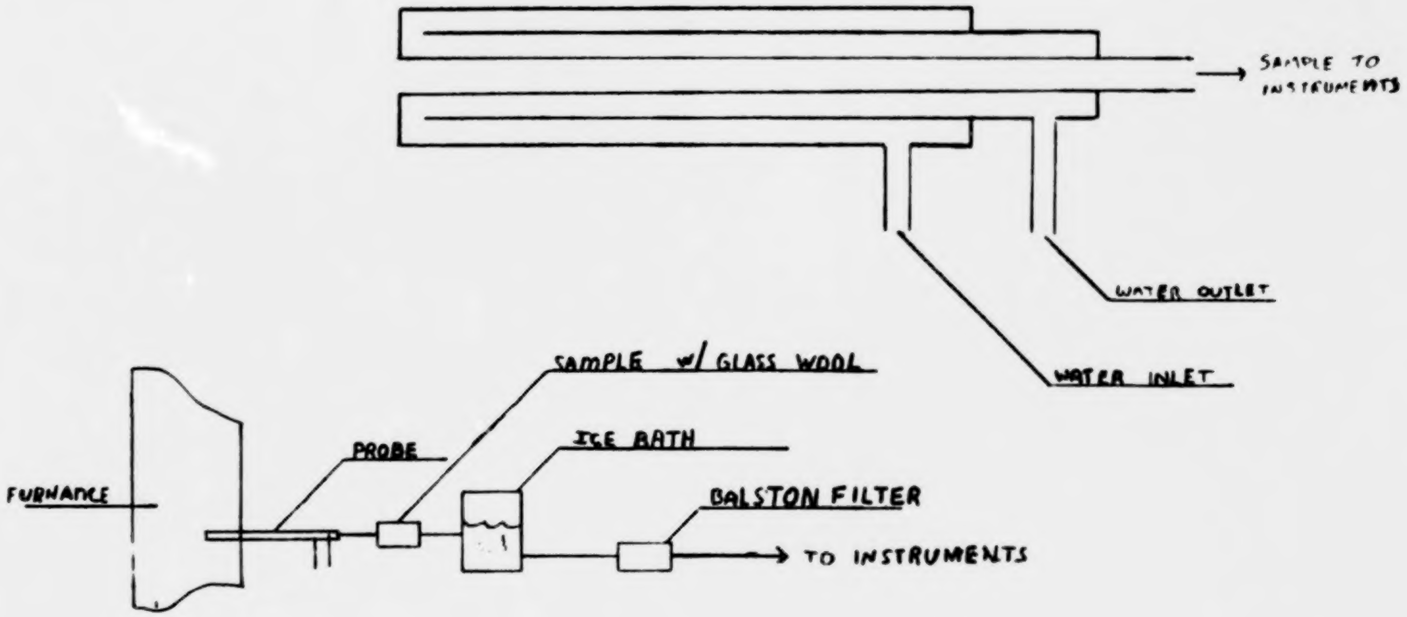

FURNACE
FILTER
FLASK FOR WATER
PUMP

WATER SPRAY PROBE

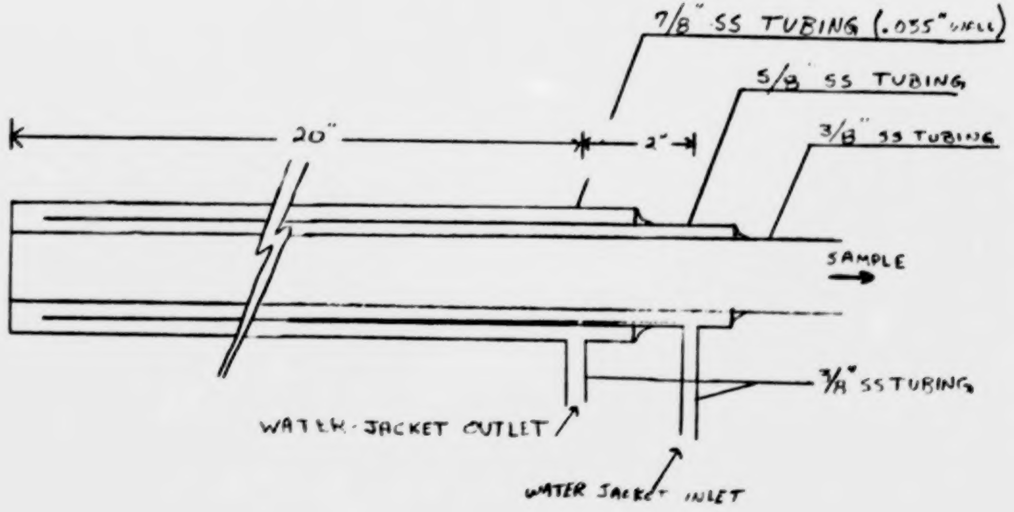

1 1/8" TUBING
7/8 TUBING
5/8 TUBING
3/8" TUBING
4-1/32" HOLES
SPRAY H_2O INLET
WATER JACKET INLET
WATER JACKET OUTLET

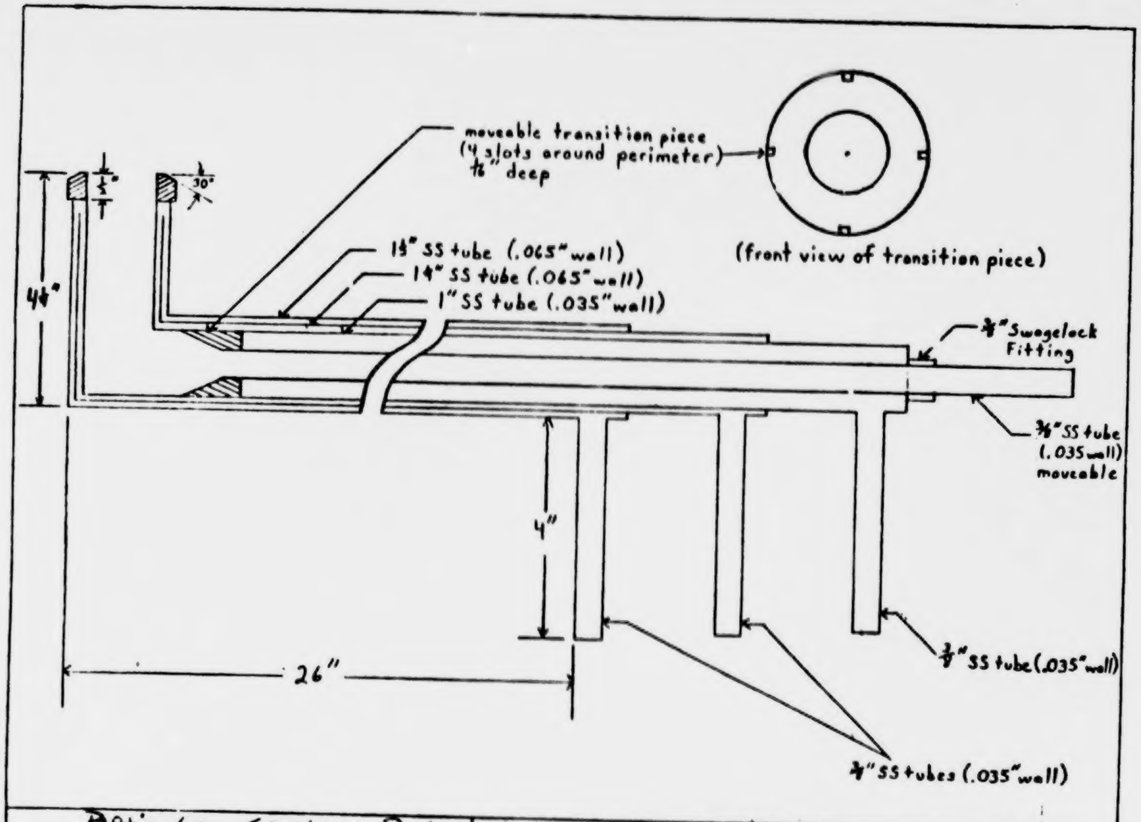
* NOTE: WATER SPRAY PROBE USED AS PARTICULATE SAMPLING PROBE

FLUE GAS SAMPLING PROBE


SAMPLE TO INSTRUMENTS
WATER OUTLET
WATER INLET
SAMPLE w/ GLASS WOOL
PROBE
ICE BATH
FURNANCE
BALSTON FILTER
TO INSTRUMENTS

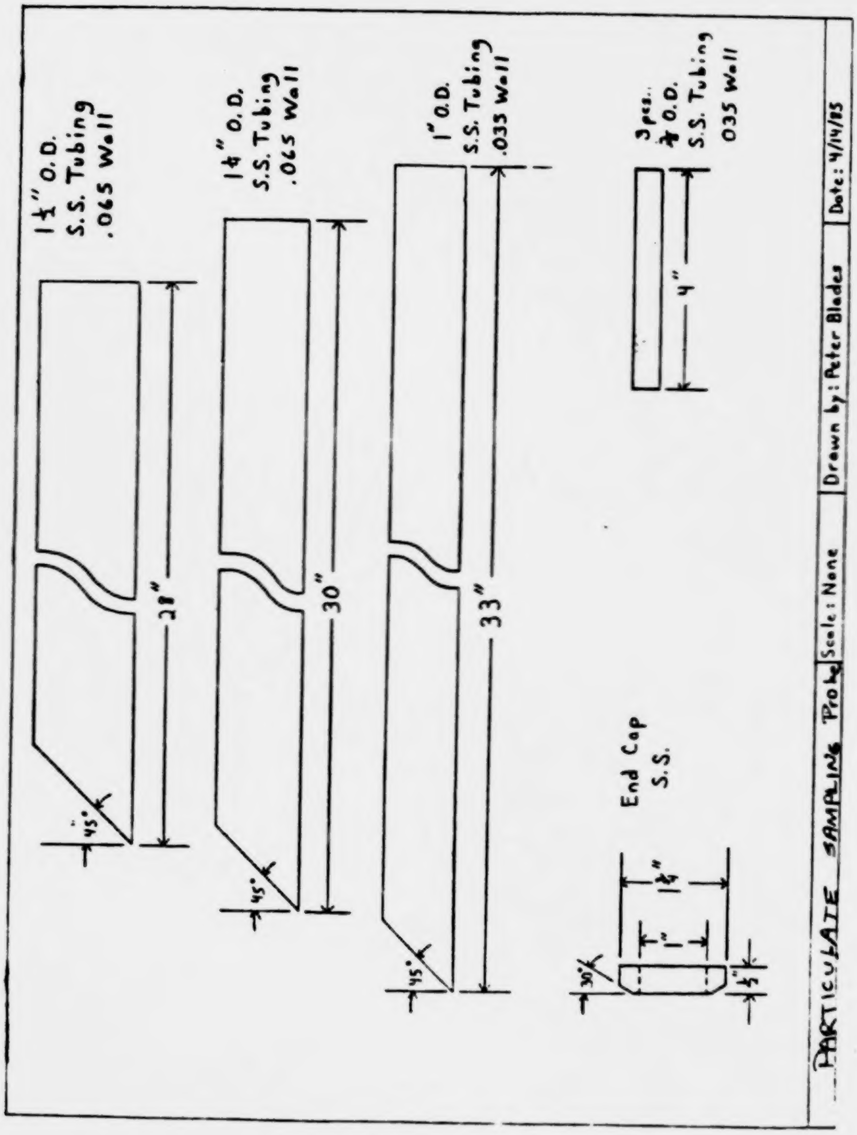
WATER-JACKETED PROBE

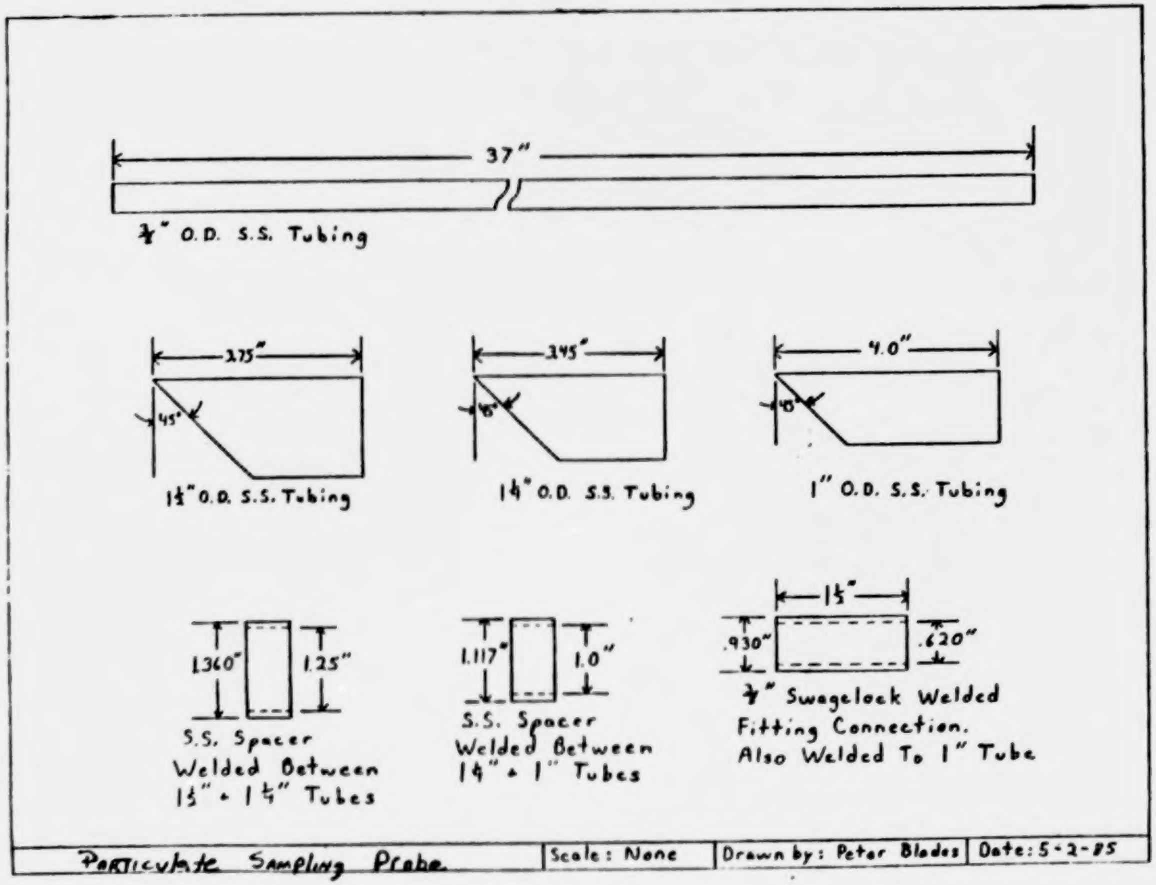

7/8" SS TUBING (.035" wall)
5/8" SS TUBING
3/8" SS TUBING
20"
2"
SAMPLE
3/8" SS TUBING
WATER-JACKET OUTLET
WATER JACKET INLET



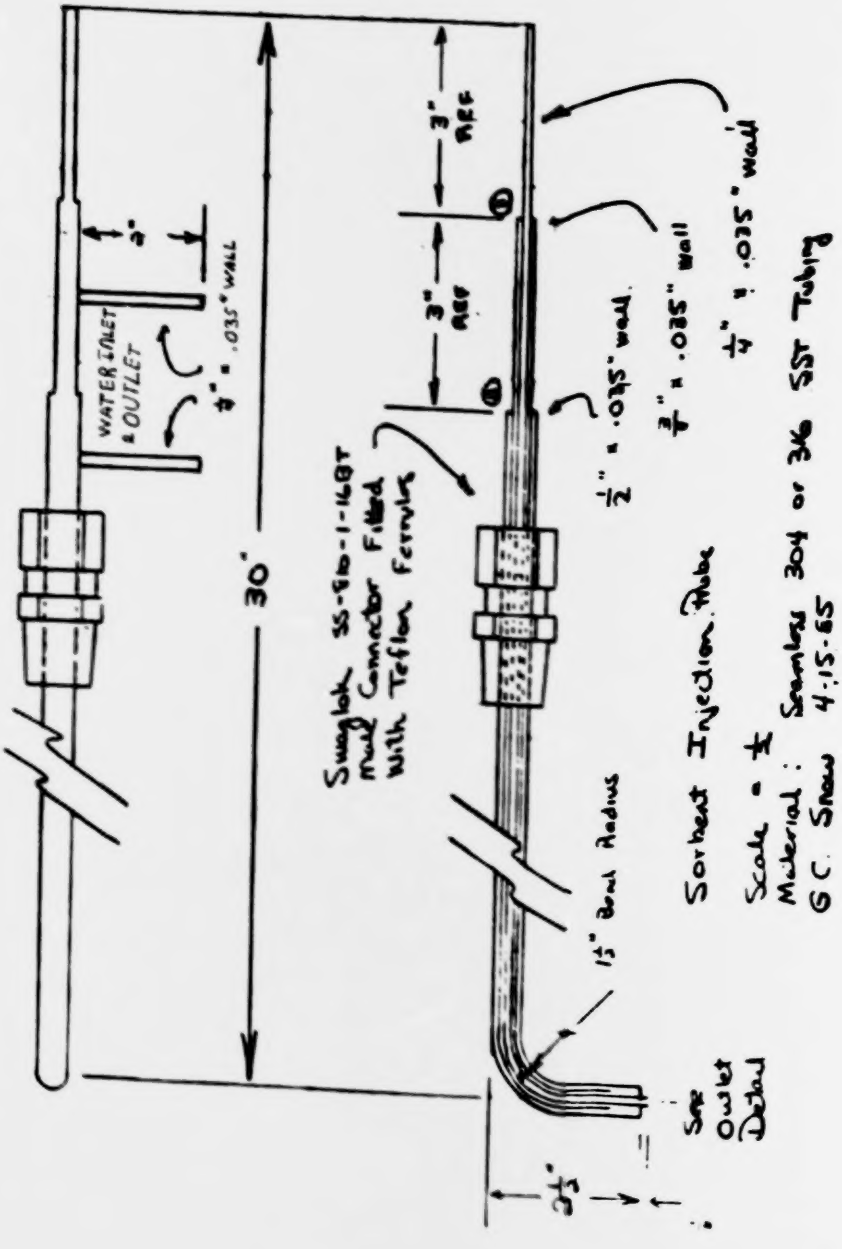
moveable transition piece
(4 slots around perimeter)
1/16" deep
(front view of transition piece)
1½" SS tube (.065" wall)
1¼" SS tube (.065" wall)
1" SS tube (.035" wall)
3/8" Swagelock Fitting
3/16" SS tube (.035 wall) moveable
3/8" SS tube (.035" wall)
1/4" SS tubes (.035" wall)
4¼"
½"
30°
4"
26"

PARTICULATE SAMPLING Probe	Scale: None	Drawn by: Peter Blades	Date: 4/14/85


1½" O.D.
S.S. Tubing
.065 Wall
1¼" O.D.
S.S. Tubing
.065 Wall
1" O.D.
S.S. Tubing
.035 Wall
3 pcs.
¼ O.D.
S.S. Tubing
035 Wall
28"
30"
33"
4"
45°
45°
45°
End Cap
S.S.
30°
1"
1¼"
¼"
PARTICULATE SAMPLING Probe
Scale: None
Drawn by: Peter Blades
Date: 4/14/85


37"
⅞" O.D. S.S. Tubing
2.75"
45°
1½" O.D. S.S. Tubing
3.45"
45°
1¼" O.D. S.S. Tubing
4.0"
45°
1" O.D. S.S. Tubing
1.360"
1.25"
S.S. Spacer
Welded Between
1½" + 1¼" Tubes
1.117"
1.0"
S.S. Spacer
Welded Between
1¼" + 1" Tubes
1½"
.930"
.620"
⅞" Swagelock Welded
Fitting Connection.
Also Welded To 1" Tube

Particulate Sampling Probe | Scale: None | Drawn by: Peter Blades | Date: 5-2-85


WATER INLET & OUTLET
1/4" x .035" WALL
1/2"
30"
3" REF
3" REF
①
①
Swagelok SS-810-1-16BT Male Connector Filled With Teflon Ferrules
1/2" x .035" wall
3/8" x .035" wall
1/4" x .035" wall
1 1/2" Bend Radius
See Outlet Detail
2 1/2"

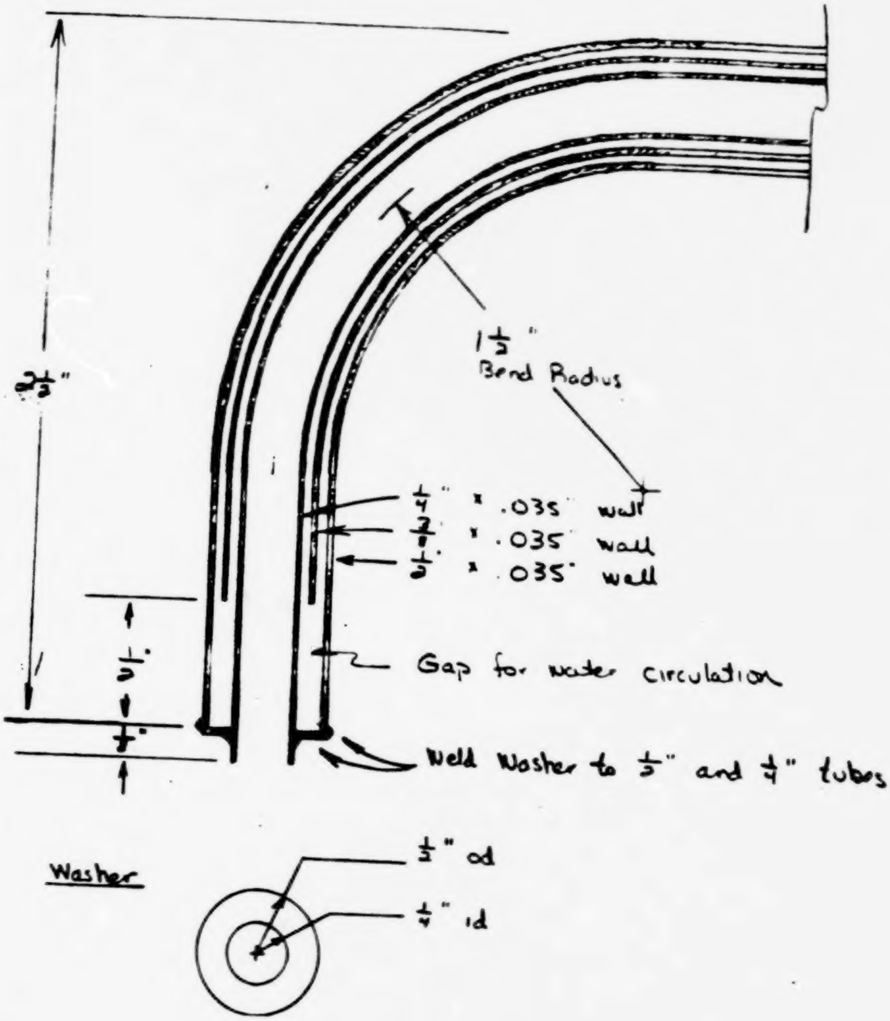
Sorbent Injection Probe

Scale = 1/2
Material: Seamless 304 or 316 SST Tubing
G.C. Snow 4-15-85

Sorbent Injection Probe
Outlet Detail

G C Snow
4-12-85

Scale = Double


2½"
1½"
Bend Radius
¼" x .035" wall
⅜" x .035" wall
½" x .035" wall
½"
⅛"
Gap for water circulation
Weld Washer to ½" and ¼" tubes
Washer
½" od
¼" id

G. C. Snow
4-15-85

Sorbent Injection Probe

Order Of Assembly

① Cut tubes to approximate lengths

$\frac{1}{2}$" tube	32"
$\frac{3}{8}$" tube	28 $\frac{1}{4}$"
$\frac{1}{4}$" tube	36"

② Drill hole on one wall of $\frac{1}{2}$" and $\frac{3}{8}$" tube to accept $\frac{1}{4}$" water inlet and outlet tubes

③ Slip $\frac{1}{2}$", $\frac{3}{8}$", and $\frac{1}{4}$" tubes together with 3" end spacings shown on assembly drawing

④ Make concentric welds at Ⓐ & Ⓑ

⑤ Install Swaglok male connector with Teflon ferrules (pipe threads away from Ⓐ & Ⓑ)

⑥ Fit $\frac{1}{2}$" tube bender (1$\frac{1}{2}$" bend radius) and make 90° bend at location shown on assembly drawing. Note, location of bender is critical to assure correct overall probe length and correct gap spacing of $\frac{3}{8}$" tube as shown in Outlet Detail Drawing.

⑦ Cut off $\frac{1}{2}$" and $\frac{1}{4}$" tubes to give 2$\frac{1}{2}$" and $\frac{1}{8}$" dimensions as shown on drawings

Continued

⑤

⑧ Dress inside of $\frac{1}{4}$" tube to remove burrs and edges

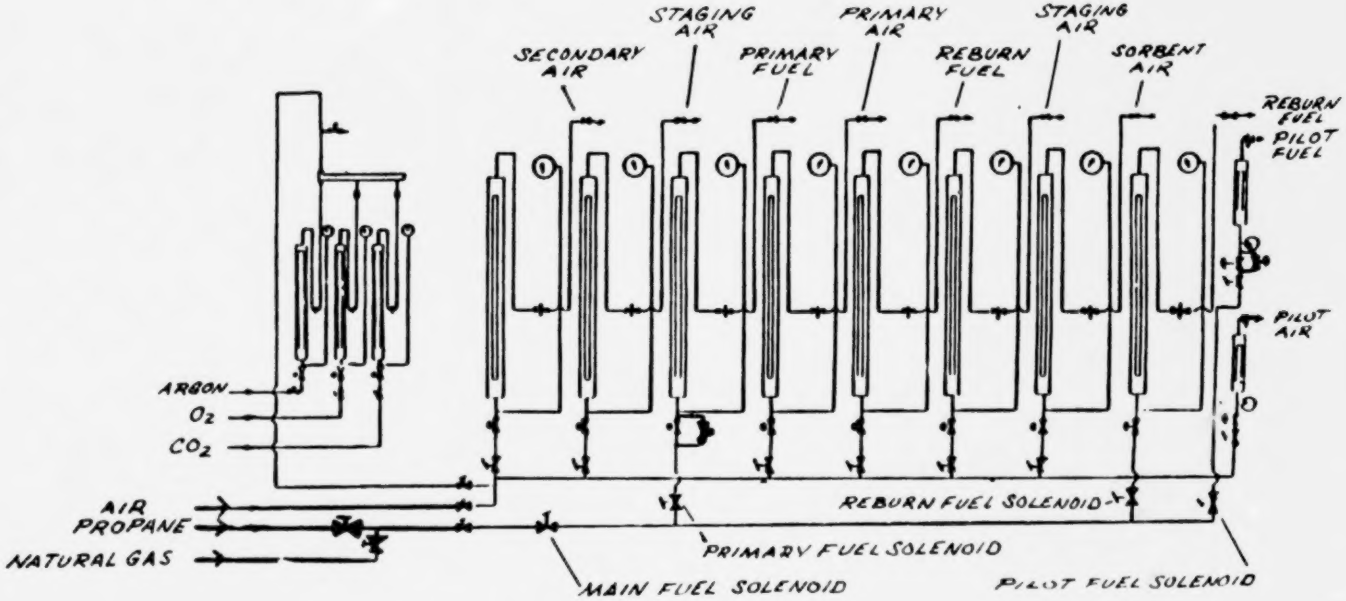
⑨ Weld washer to $\frac{1}{2}$" and $\frac{1}{4}$" tubes to center $\frac{1}{4}$" tube and provide water tight seal

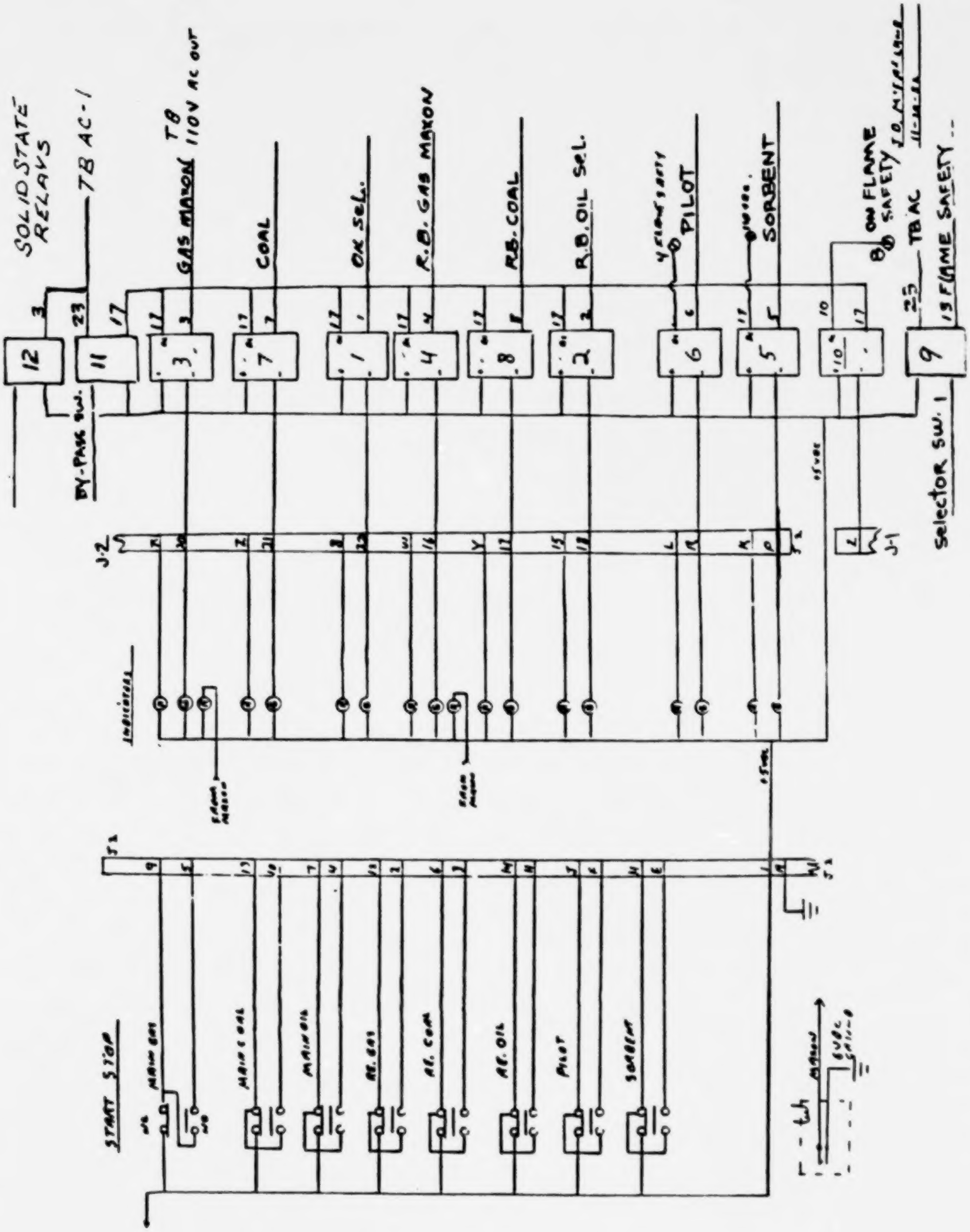
⑩ Weld on water inlet and outlet tubes

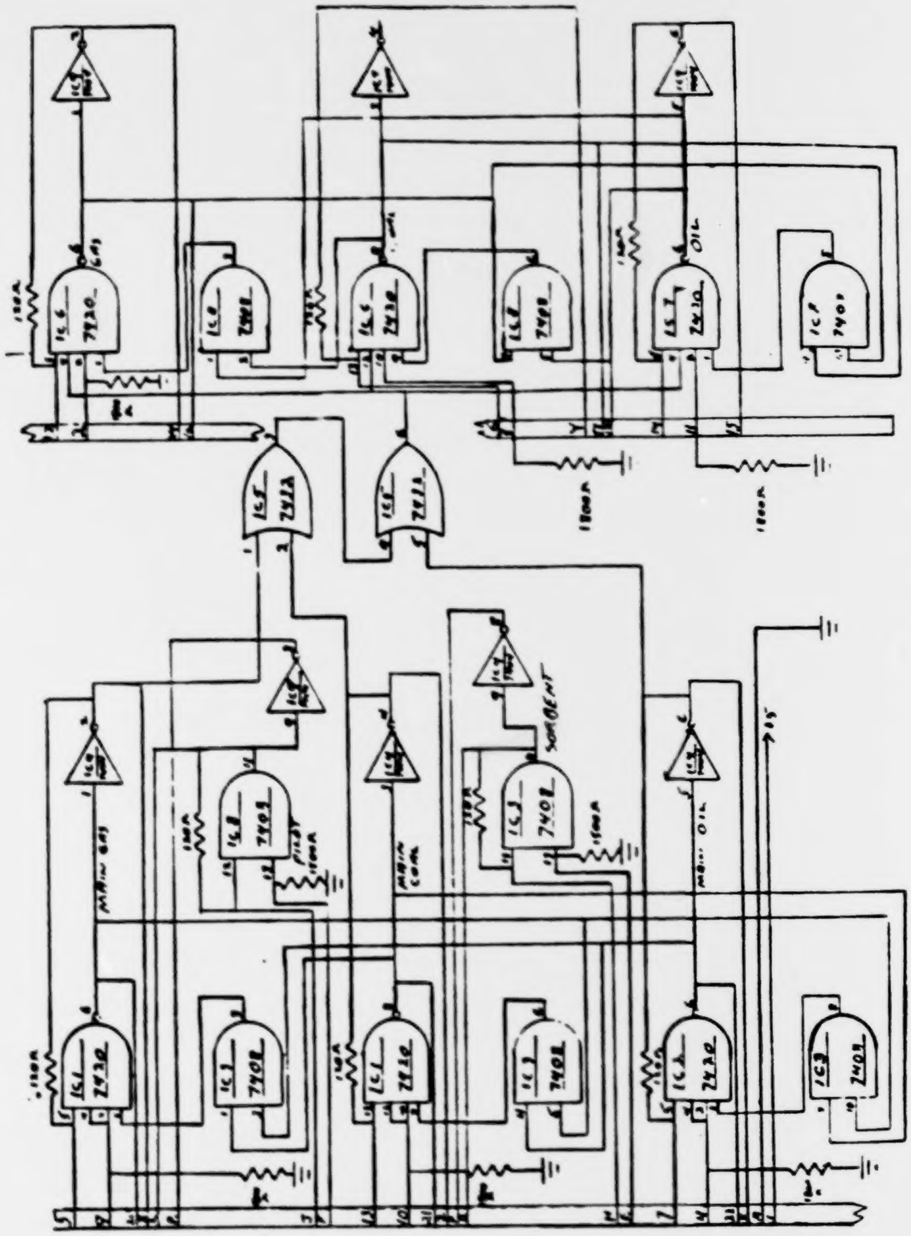
APPENDIX A.4

AIR AND FUEL FEED CONTROLS AND ELECTRICAL SCHEMATICS

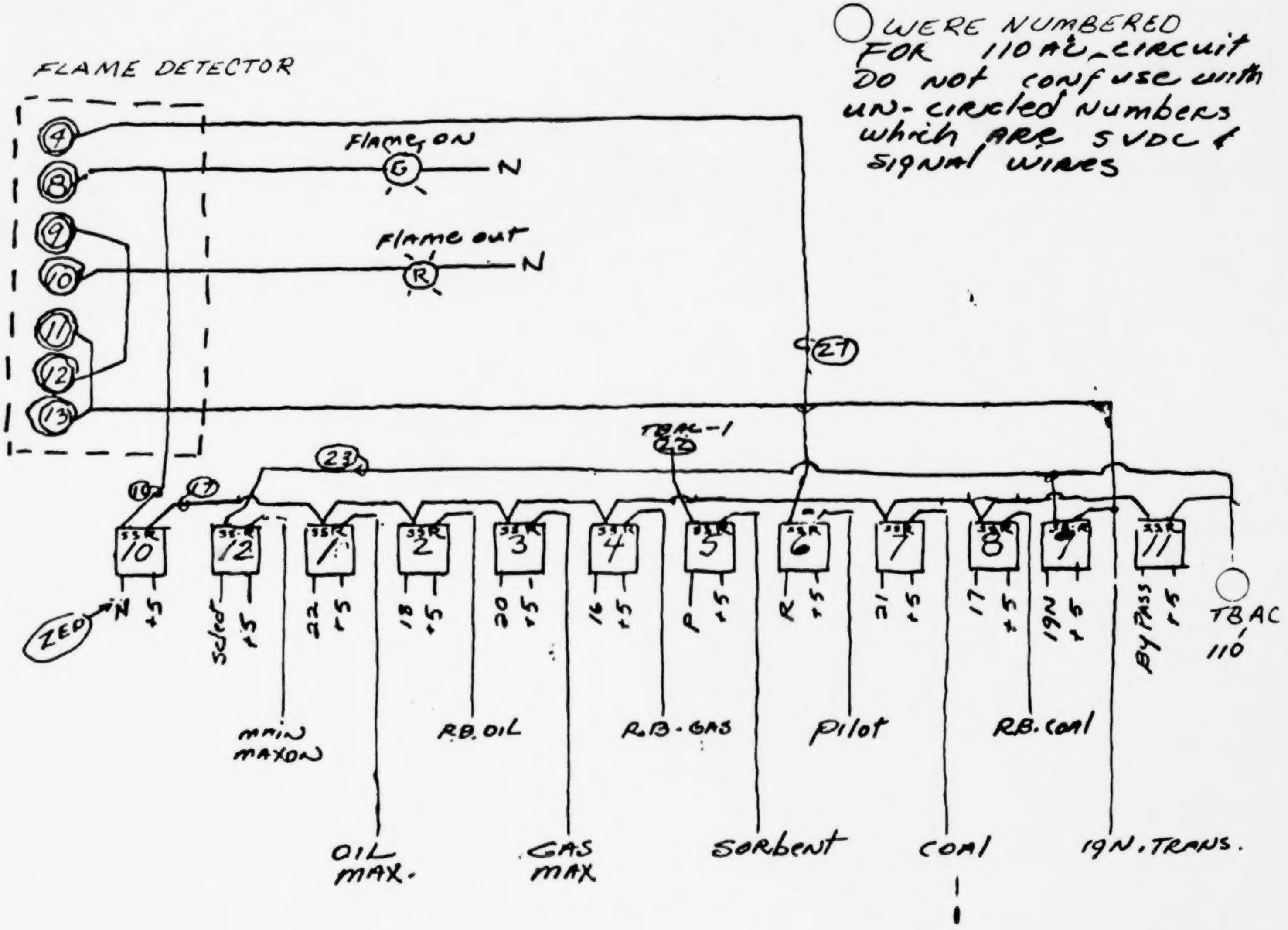
AIR AND FUEL ROTAMETERS


SECONDARY AIR
STAGING AIR
PRIMARY FUEL
PRIMARY AIR
REBURN FUEL
STAGING AIR
SORBENT AIR
REBURN FUEL
PILOT FUEL
PILOT AIR
ARGON
O_2
CO_2
AIR
PROPANE
NATURAL GAS
REBURN FUEL SOLENOID
PRIMARY FUEL SOLENOID
MAIN FUEL SOLENOID
PILOT FUEL SOLENOID


SOLID STATE RELAYS
TB AC-1
GAS MAXON
TB 110V AC OUT
COAL
OX SEL.
R.B. GAS MAXON
R.B. COAL
R.B. OIL SEL.
PILOT
SORBENT
ON FLAME SAFETY
TB AC
FLAME SAFETY
BY-PASS SW.
SELECTOR SW. 1
12
11
3
7
1
4
8
2
6
5
10
9
J-2
J-1
START STOP
MAIN GAS
MAIN COAL
MAIN OIL
RB. GAS
RB. COAL
RB. OIL
PILOT
SORBENT
INDICATORS



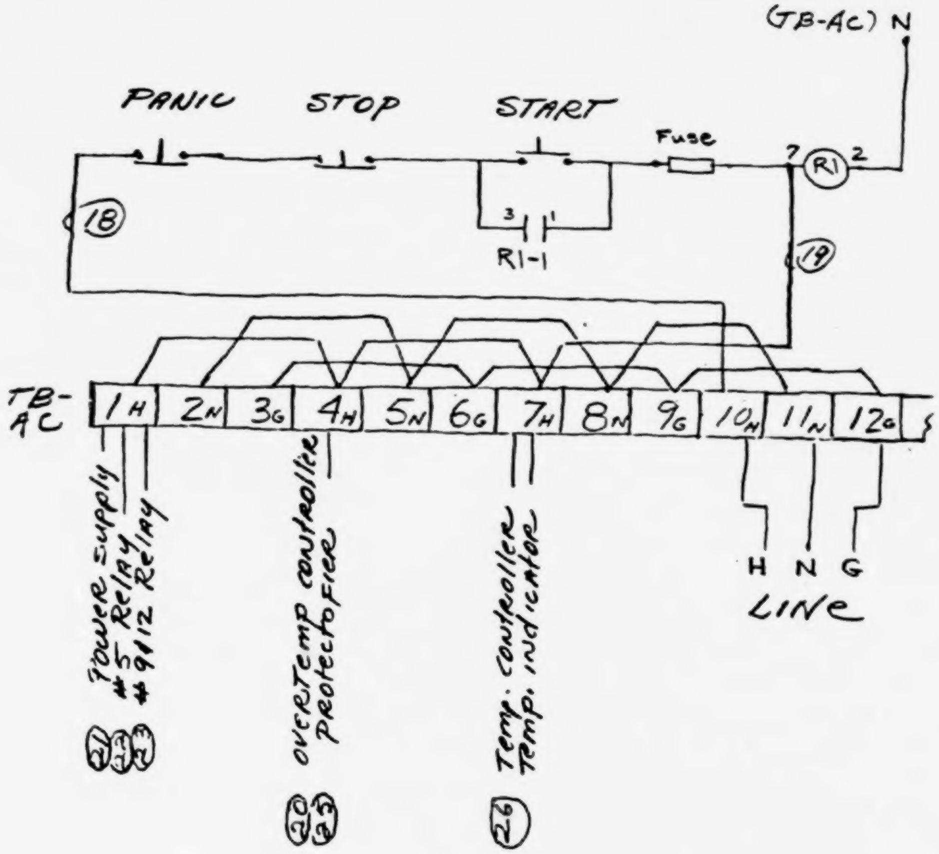
GAS
OIL
SORBENT
MAIN GAS
MAIN COAL
MAIN OIL
7430
7408
7432
7404

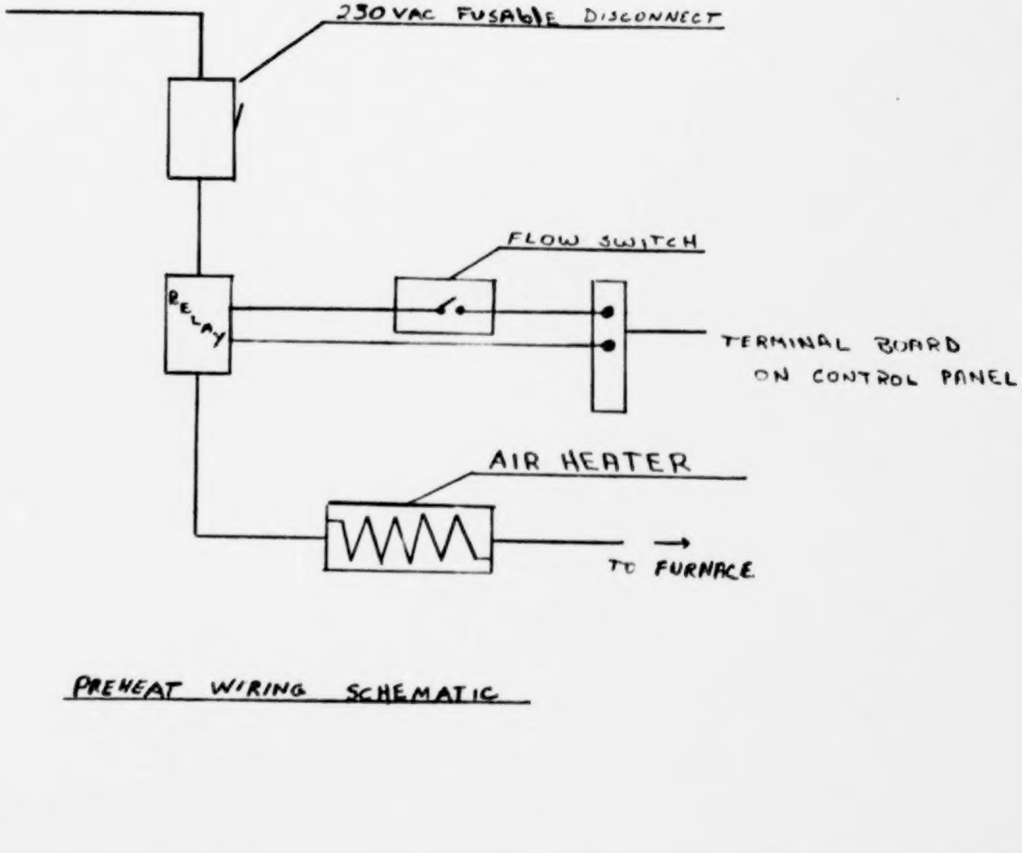


FLAME DETECTOR
4
8
9
10
11
12
13
Flame on
G
N
Flame out
R
N
27
23
TBAC-1
22
10
17
SSR
10
12
1
2
3
4
5
6
7
8
9
11
ZED
N
+5
Select
+5
22
+5
18
+5
30
+5
16
+5
P
+5
R
+5
31
+5
17
+5
19N
+5
By Pass
+5
TBAC
110
main
maxon
OIL
MAX.
R.B. OIL
GAS
MAX
R.B. GAS
SORbent
Pilot
COAL
R.B. COAL
IGN. TRANS.

○ WERE NUMBERED FOR 110 AC CIRCUIT DO NOT CONFUSE with UN-CIRCLED Numbers which ARE 5 VDC & SIGNAL WIRES

E.P.A. SAFETY SYSTEM

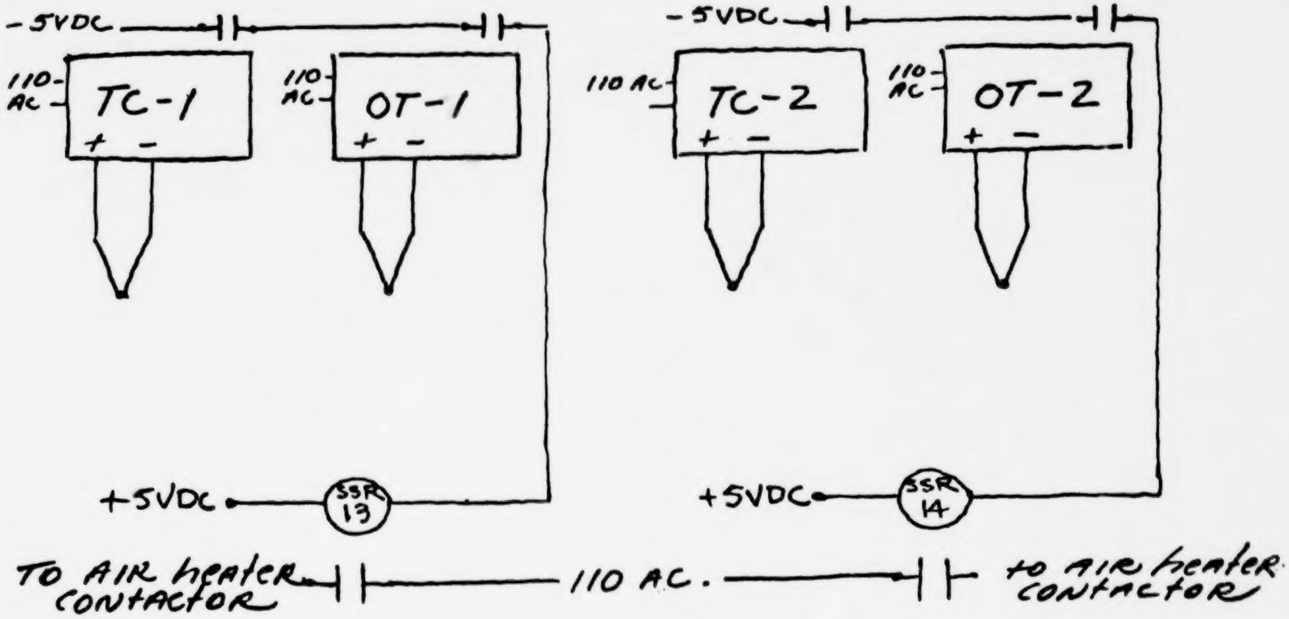

(TB-AC) N
PANIC
STOP
START
Fuse
7
R1
2
3
1
R1-1
18
19
TB-AC
1H
2N
3G
4H
5N
6G
7H
8N
9G
10H
11N
12G
Power supply
#5 Relay
#9&12 Relay
21
22
23
overtemp controller
Protectofier
20
25
Temp. controller
Temp. indicator
26
H
N
G
LINE



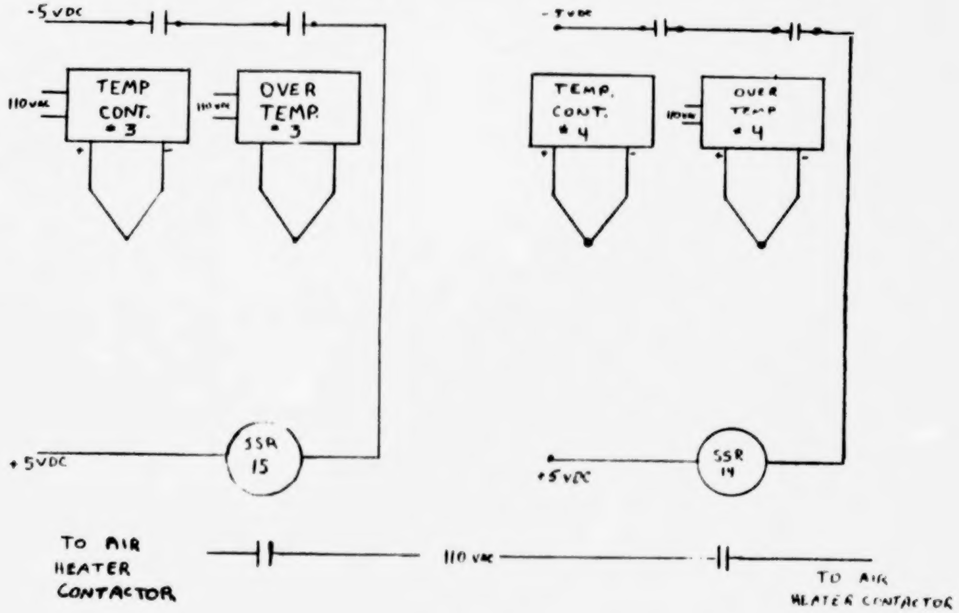
230 VAC FUSABLE DISCONNECT
RELAY
FLOW SWITCH
TERMINAL BOARD ON CONTROL PANEL
AIR HEATER
TO FURNACE

PREHEAT WIRING SCHEMATIC

EPA SAFETY SYSTEM AND TEMPERATURE CONTROL


-5VDC
110-AC
TC-1
+ -
110-AC
OT-1
+ -
-5VDC
110 AC
TC-2
+ -
110-AC
OT-2
+ -
+5VDC
SSR 13
+5VDC
SSR 14
TO AIR heater CONTACTOR
110 AC.
to AIR heater CONTACTOR

EPA SAFETY SYSTEM FOR TEMPERATURE CONTROL 3/4


-5VDC
110VAC
TEMP CONT. #3
110VAC
OVER TEMP. #3
+
-
+5VDC
SSR 15
-5VDC
TEMP. CONT. #4
110VAC
OVER TEMP #4
+
-
+5VDC
SSR 14
TO AIR HEATER CONTACTOR
110VAC
TO AIR HEATER CONTACTOR

APPENDIX B

RAW DATA

Partial Listing from Demonstration Sorbent Testing

Date	Run Number	Fuel	Sorbent	Firing Rate (Btu/hr) (kW)	Fuel Flow (~~lbm~~/hr)	Air Flow (SCFm)	SO_2 Flow (SCFm) $\times 10^{-2}$	SR Determined From Flows	Sorbent Flow (lbm/hr)	$\frac{Ca}{S}$ (molar)	% Capture @ $\frac{Ca}{S} = 2$
7-29-85	2B8A0C 1	N. Gas	Mercer	47300 14	(SCFm) 0.765	9.30	1.82	1.28	0 .355	0 1.55	51.5
	2B8A0C 2							1.28	0 .355	0 1.55	54.1
	2B8A0C 3							1.28	0 .355	0 1.55	58.7
	2B8A0C 4							1.28	0 .355	0 1.55	48.5
											53.2 s = 4.3 (8.1%)

②

From flows

Date	Run Number	O_2 (%)	CO_2 (%)	NO (ppm)	SO_2 (ppm)	SO_2 at 0% O_2 (ppm)	SO_2 Removed (%)	SR Calculated From Digistrip	O_2 Predicted (%)	CO_2 Predicted (%)	SO_2 Predicted (ppm)
7-24-85	2B8A0C	5.16	8.90		1687	2236	–	1.29	5.0	8.9	2120
	1	5.12	9.04		1016	1344	39.9	1.29			
	2B8A0C	5.31	8.76		1585	2121	-	1.31	5.0	8.9	2120
	2	4.91	9.00		944	1232	41.9	1.28			
	2B8A0C 3	4.97	9.00		1620	2122	-	1.28	5.0	8.9	2120
		4.42	8.26		913	1156	45.5	1.24			
	2B8A0C 4	5.17	8.98		1576	2091	-	1.29	5.0	8.9	2120
		5.15	8.98		984	1304	37.6	1.29			

①

Date	Run Number	Fuel	Sorbent	Firing Rate (Btu/hr) (kW)	Fuel Flow $\left(\frac{lbm}{hr}\right)$	Air Flow (scfm)	SO_2 Flow (scfm) $\times 10^{-2}$	SR Determined From Flows	Sorbent Flow $\left(\frac{lbm}{hr}\right)$	$\frac{Ca}{S}$ (molar)	% Capture @ $\frac{Ca}{S} = 2$
7 31 85	3B8AϕC 1	Pitts 8	Mercer	47200 14	3.50	9.76	–	1.24	0 .467	0 2.08	57.3
	3B8AϕC 2								0 .467	0 2.08	58.5
	3B8AϕC 3								0 .467	0 2.08	61.5
	3B8AϕC 4								0 .467	0 2.08	62.5
	3B8AϕC 5								0 .467	0 2.08	56.7
	3B8AϕC 6								0 .467	0 2.08	59.2
											59.3 σ = 2.3 (3.9%)

②

from flows

Date	Run Number	O_2 (%)	CO_2 (%)	NO (ppm)	SO_2 (ppm)	SO_2 at 0% O_2 (ppm)	SO_2 Removed (%)	SR Calculated From Digistrip	O_2 Predicted (%)	CO_2 Predicted (%)	SO_2 Predicted (ppm)
7-31-85	3B8AØC	4.07	13.4		2024	2510	–	1.23	4.00	14.6	1903
	1	4.08	13.4		817	1014	59.6	1.23			
	3B8AØC	4.14	13.4		1966	2449	–	1.24	4.00	14.6	1903
	2	4.54	13.0		752	960	60.8	1.26			
	3B8AØC	4.67	13.0		1863	2396	–	1.27	4.00	14.6	1903
	3	4.82	12.8		665	863	64.0	1.28			
	3B8AØC	4.77	12.9		1837	2376	–	1.28	4.00	14.6	1903
	4	5.08	12.6		630	831	65.0	1.31			
	3B8AØC	4.24	13.4		1914	2397	–	1.24	4.00	14.6	1903
	5	4.28	13.3		784	984	58.9	1.25			
	3B8AØC	4.57	13.1		1866	2385	–	1.27	4.00	14.6	1903
	6	4.58	13.1		717	917	61.6	1.27			

①

Date	Run Number	Fuel	Sorbent	Firing Rate (Btu/hr) (kW)	Fuel Flow $\left(\frac{lbm}{hr}\right)$	Air Flow (scfm)	SO_2 Flow (scfm) $\times 10^{-2}$	SR Determined From Flows	Sorbent Flow $\left(\frac{lbm}{hr}\right)$	$\frac{Ca}{S}$ (molar)	% Capture @ $\frac{Ca}{S} = 2$
8-5-85	3B9AØC 1	P.H 8	Kemikal	47200 14	3.50	9.76	–	1.24	0 .450	0 1.94	58.8
	3B9AØC 2								0 .450	0 1.94	61.0
	3B9AØC 3								0 .450	0 1.94	64.1
	3B9AØC 4								0 .450	0 1.94	64.0
											62.0 σ = 2.6 (4.1%)

②

from flows

Date	Run Number	O_2 (%)	CO_2 (%)	NO (ppm)	SO_2 (ppm)	SO_2 at 0% O_2 (ppm)	SO_2 Removed (%)	SR Calculated From Digistrip	O_2 Predicted (%)	CO_2 Predicted (%)	SO_2 Predicted (ppm)
8-5-85	3B9A0C	4.07	14.0		2149	2666	–	1.23	4.00	14.6	1903
	1	3.64	14.2	445	947	1145	57.1	1.20			
	3B9A0C	3.79	14.3	428	2077	2534	–	1.21	4.00	14.6	1903
	2	3.72	14.3	431	851	1034	59.2	1.21			
	3B9A0C	3.93	14.2	433	2069	2545	–	1.22	4.00	14.6	1903
	3	3.91	14.2	438	783	962	62.2				
	3B9A0C	4.05	14.1	443	2034	2520	–	1.23	4.00	14.6	1903
	4	4.03	14.1	451	772	955	62.1	1.23			

①

Date	Run Number	Fuel	Sorbent	Firing Rate (Btu/hr) (kW)	Fuel Flow (SCFM)	Air Flow (SCFM)	SO_2 Flow (SCFM) $\times 10^{-2}$	SR Determined From Flows	Sorbent Flow $\left(\frac{lbm}{hr}\right)$	$\frac{Ca}{S}$ (molar)	% capture @ $\frac{Ca}{S} = 2$
8-6-85	2B9A0C 1	N. Gas	Kemikal	47300 14	0.765	9.30	1.82	1.28	0 .450	0 1.90	36.6
	2B9A0C 2								0 .450	0 1.90	35.7
	2B9A0C 3								0 .450	0 1.90	34.4
	2B9A0C 4								0 .450	0 1.90	35.3
											35.5 s = .91 (2.6%)

③

from flows

Date	Run Number	O_2 (%)	CO_2 (%)	NO (ppm)	SO_2 (ppm)	SO_2 at 0% O_2 (ppm)	SO_2 Removed (%)	SR Calculated From Digistrip	O_2 Predicted (%)	CO_2 Predicted (%)	SO_2 Predicted (ppm)
8.6.85	289A0C 1	5.02 5.07	9.10 9.05	60 60	1914 1245	2515 1641	- 34.8	1.28 1.29	5.0	8.9	2120
	289A0C 2	4.94 4.86	9.32 9.29		1950 1295	2550 1685	- 33.9	1.28 1.27	5.0	8.9	2120
	289A0C 3	4.90 4.85	9.29 9.28		1950 1316	2543 1712	- 32.7	1.28	5.0	8.9	2120
	289A0C 4	4.91 4.84	9.26 9.28		1971 1315	2572 1709	- 33.6	1.28 1.27	5.0	8.9	2120

(1)

Date	Run Number	Fuel	Sorbent	Firing Rate (Btu/hr) (kW)	Fuel Flow (scfm)	Air Flow (scfm)	SO_2 Flow (scfm) $\times 10^{-2}$	SR Determined From Flows	Sorbent Flow $\left(\frac{lbm}{hr}\right)$	$\frac{Ca}{S}$ (molar)	% Capture @ $\frac{Ca}{S} = 2$
8-8-85	2810A0C 1	N. Gas	Kemidol	47300 14	0.765	9.30	1.82	1.28	0 .780	0 2.02	68.4
	2810A0C 2								0 .780	0 2.02	63.3
	2810A0C 3								0 .780	0 2.02	63.5
	2810A0C 4								0 .780	0 2.02	62.8
											64.5 s = 2.6 (4.1%)

②

from flows

Date	Run Number	O_2 (%)	CO_2 (%)	NO (ppm)	SO_2 (ppm)	SO_2 at 0% O_2 (ppm)	SO_2 Removed (%)	SR Calculated From Digistrip	O_2 Predicted (%)	CO_2 Predicted (%)	SO_2 Predicted (ppm)
8-8-85	2B10ABC	4.81	10.3		1846	2395	-	1.27	5.0	8.9	2120
	1	4.75	10.3		572	734	69.1	1.27			
	2B10ABC	4.82	10.3		1723	2236	-	1.27	5.0	8.9	2120
	2	4.76	10.2		625	808	63.9	1.27			
	2B10ABC	4.76	10.3		1814	2345	-	1.27	5.0	8.9	2120
	3	4.76	10.2		652	843	64.1	1.27			
	2B10ABC	4.82	10.2		1711	2221	-	1.27	5.0	8.9	2120
	4	4.68	10.2		651	812	63.4	1.26			

①

Date	Run Number	Fuel	Sorbent	Firing Rate (Btu/hr) (kW)	Fuel Flow $\left(\frac{lbm}{hr}\right)$	Air Flow (scFm)	SO_2 Flow (scFm) $\times 10^{-2}$	SR Determined From Flows	Sorbent Flow $\left(\frac{lbm}{hr}\right)$	$\frac{Ca}{S}$ (molar)	% Capture @ $\frac{Ca}{S} = 2$
8-8-85	3B10A0C 1	Pitts 8	Kemidol	47200 14	3.50	9.76	–	1.24	0 .780	0 2.06	80.8
	3B10A0C 2								0 .780	0 2.06	81.3
	3B10A0C 3								0 .780	0 2.06	79.7
	3B10A0C 4								0 .780	0 2.06	77.8
											79.7 s=1.55 (1.9%)

②

from flows

Date	Run Number	O_2 (%)	CO_2 (%)	NO (ppm)	SO_2 (ppm)	SO_2 at 0% O_2 (ppm)	SO_2 Removed (%)	SR Calculated From Digistrip	O_2 Predicted (%)	CO_2 Predicted (%)	SO_2 Predicted (ppm)
8-8-85	3B10A0C 1	4.30 4.08	16.3 16.5		1912 330	2404 410	- 83.0	1.26 1.24	4.00	14.6	1903
	3B10A0C 2	4.14 4.89	16.5 15.5		1849 296	2366 386	- 83.7	1.25 1.30	4.00	14.6	1903
	3B10A0C 3	4.09 4.34	16.5 16.2		1845 335	2353 422	- 82.1	1.24 1.26	4.00	14.6	1903
	3B10A0C 4	4.17 4.14	16.5 16.4		1915 379	2369 472	- 80.1	1.25 1.25	4.00	14.6	1903

(i)

Date	Run Number	Fuel	Sorbent	Firing Rate (Btu/hr) (kW)	Fuel Flow (scfm)	Air Flow (scfm)	SO_2 Flow (scfm) $\times 10^{-2}$	SR Determined From Flows	Sorbent Flow $\left(\frac{lbm}{hr}\right)$	$\frac{Ca}{S}$ (molar)	% Capture @ $\frac{Ca}{S}$: 2
	2B11AΦC 1.	N. Gas	Ivory	47300 14	.765	9.30	1.82	1.28	0 .785	0 1.94	72.6
	2B11AΦC 2								0 .785	0 1.94	64.5
8 14-85	2B11AΦC 1								0 .785	0 1.94	67.5
	2B11AΦC. 2								0 .785	0 1.94	69 8
	2B11AΦC 3								0 .785	0 1.94	63 2
	2B11AΦC. 4								0 .785	0 1.94	63 8
											66.9 s= 3.8 (5.6%)

②

from flows

Date	Run Number	O_2 (%)	CO_2 (%)	NO (ppm)	SO_2 (ppm)	SO_2 at 0% O_2 (ppm)	SO_2 Removed (%)	SR Calculated From Digistrip	O_2 Predicted (%)	CO_2 Predicted (%)	SO_2 Predicted (ppm)
	2B11A0C 1	5.02 4.98	9.34 9.28		1646 490	2163 642	- 70.3	1.29 1.28	5.0	8.9	2120
	2B11A0C 2	5.01 4.98	9.32 9.28		1963 737	2578 966	- 62.5	1.28 1.28	5.0	8.9	2120
8-14-85	2B11A0C 1	5.02 4.96	9.35 9.35		1799 625	2364 818	- 65.4	1.29 1.28	5.0	8.9	2120
	2B11A0C 2	5.03 4.98	9.35 9.34		1781 577	2342 757	- 67.7	1.29 1.28	5.0	8.9	2120
	2B11A0C 3	4.97 4.93	9.39 9.38		1986 772	2602 1009	- 61.2	1.28 1.28	5.0	8.9	2120
	2B11A0C 4	4.98 4.92	9.34 9.38		2024 776	2653 1013	- 61.8	1.28 1.28	5.0	8.9	2120

①

Date	Run Number	Fuel	Sorbent	Firing Rate (Btu/hr) (kW)	Fuel Flow $\left(\frac{lbm}{hr}\right)$	Air Flow (SCFm)	SO_2 Flow (SCFm) $\times 10^{-2}$	SR Determined From Flows	Sorbent Flow $\left(\frac{lbm}{hr}\right)$	$\frac{Ca}{S}$ (molar)	% Capture @ $\frac{Ca}{S} = 2.5$
9-11-85	3B10A0 1	Atls 3	Kemidol	47700 14	3.50	9.76	-	1.24	0 .986	0 2.60	80.8
	3B10A0 2								0 .986	0 2.60	80.6
	3B10A0 3								0 .986	0 2.60	80.0
	3B10A0 4								0 .986	0 2.60	79.3
											80.2 s = .68 (.84 %)

from flows

Date	Run Number	O_2 (%)	CO_2 (%)	NO (ppm)	SO_2 (ppm)	SO_2 at 0% O_2 (ppm)	SO_2 Removed (%)	SR Calculated From Digistrip	O_2 Predicted (%)	CO_2 Predicted (%)	SO_2 Predicted (ppm)
9-11-85	3B10A0 1	4.00 3.85	14.8 14.8		1987 320	2454 392	– 84.0	1.23 1.22	4.00	14.6	1903
	3B10A0 2	3.91 4.04	14.8 14.6		1934 310	2376 384	-- 83.8	1.22 1.23	4.00	14.6	1903
	3B10A0 3	3.86 3.13	14.9 14.7		1978 352	2424 433	– 82.1	1.22 1.22	4.00	14.6	1903
	3B10A0 4	3.89 3.96	14.8 14.7		1985 346	2436 426	– 82.5	1.22 1.23	4.00	14.6	1903

①

Date	Run Number	Fuel	Sorbent	Firing Rate (Btu/hr) (kW)	Fuel Flow $\left(\frac{lbm}{hr}\right)$	Air Flow (scfm)	SO_2 Flow (scfm) $\times 10^{-2}$	SR Determined From Flows	Sorbent Flow $\left(\frac{lbm}{hr}\right)$	$\frac{Ca}{S}$ (molar)	% Capture @
9-11-85	3B10A0C 1	Pitt 8	Kemidol	47200 14	3.50	9.76	–	1.24	0 .799	0 2.11	$\frac{Ca}{S}$: 2 74.6
	3B10A0C 2								0 .799	0 2.11	72.8
	3B10A0C 3								0 .799	0 2.11	72.0
											73.1 s= 1.3 (1.8%)
	3B10A0C 1				3.50	9.76		1.24	0 .613	0 1.62	$\frac{Ca}{S}$ = 1.5 60.6
	3B10A0C 2								0 .613	0 1.62	61.9
	3B10A0C 3								0 .613	0 1.62	61.0
											61.2 s= .67 (1.1%)

②

from flows

Date	Run Number	O_2 (%)	CO_2 (%)	NO (ppm)	SO_2 (ppm)	SO_2 at 0% O_2 (ppm)	SO_2 Removal (%)	SR Calculated From Digistrip	O_2 Predicted (%)	CO_2 Predicted (%)	SO_2 Predicted (ppm)
9-11-85	3B10A0C 1	4.00 4.21	14.7 14.5		1979 416	2444 520	— 78.7	1.23	4.00	14.6	1903
	3B10A0C 2	4.23 3.94	14.5 14.7		1870 442	2341 544	— 76.8	1.25	4.00	14.6	1903
	3B10A0C 3	4.14 4.02	14.6 14.6		1897 459	2363 568	- 76.0	1.24 1.23	4.00	14.6	1903
	3B10A0C 1	4.23 4.17	14.5 14.5		1904 662	2384 826	— 65.4	1.25 1.24	4.00	14.6	1903
	3B10A0C 2	4.29 4.29	14.5 14.5		1899 629	2387 790	— 669	1.25 1.25	4.00	14.6	1903
	3B10A0C 3	4.27 4.30	14.5 14.4		1869 636	2346 800	— 659	1.25	4.00	14.6	1903

Date	Run Number	Fuel	Sorbent	Firing Rate (Btu/hr) (kW)	Fuel Flow $\left(\frac{lbm}{hr}\right)$	Air Flow (scFm)	SO$_2$ Flow (scFm) ×10^{-2}	SR Determined From Flows	Sorbent Flow $\left(\frac{lbm}{hr}\right)$	$\frac{Ca}{S}$ (molar)	% Capture @ $\frac{Ca}{S} = 2$
4-26-85	7B12A0C 1	Boiche Pitt 8 Demo	Marblehead	47200 14	3.64	9.92	—	1.25	0 .559	0 2	69.6
	7B12A0C 2								0 .559	0 2	68.1
	7B12A0C 3								0 .559	0 2	70.4
	7B12A0C 4								0 .559	0 2	69.1
											69.3 s = .96 (1.4%)

②

from flows

Date	Run Number	O_2 (%)	CO_2 (%)	NO (ppm)	SO_2 (ppm)	SO_2 at 0% O_2 (ppm)	SO_2 Removed (%)	SR Calculated From Digistrip	O_2 Predicted (%)	CO_2 Predicted (%)	SO_2 Predicted (ppm)
7-26-85	7B12ABC 1	4.20 4.06	14.3 14.4		2232 683	2789 847	– 69.6	1.24	4.29	14.4	2309
	7B12ABC 2	4.13 4.27	14.4 14.2		2218 701	2760 880	- 68.1	1.24 1.25	4.29	14.4	2309
	7B12ABC 3	3.84 4.10	14.5 14.3		2303 671	2818 834	– 70.4	1.22	4.29	14.4	2309
	7B12ABC 4	4.26 4.06	14.2 14.3		2226 695	2792 862	– 69.1	1.25 1.23	4.29	14.4	2309

①

Date	Run Number	Fuel	Sorbent	Firing Rate (Btu/hr) (kW)	Fuel Flow $\left(\frac{lbm}{hr}\right)$	Air Flow (scfm)	SO_2 Flow (scfm) $\times 10^{-2}$	SR Determined From Flows	Sorbent Flow $\left(\frac{lbm}{hr}\right)$	$\frac{Ca}{S}$ (molar)	
9 29-85	7B10A&C	Beche Pitt 8 Demo	Kemidol	47200 14	3.64	9.92	—	1.25			82.8
											84.1
											82.5
											80.9
											82.6 s= 1.3 (16.2)

APPENDIX C

INDUCTIVELY COUPLED ARGON PLASMA EMISSION SPECTROSCOPY REPORTS

Page

C.1 Analysis of Calcium Silicate (Portland Cement) Sorbents C-2

C.2 Analysis of Demonstration Sorbents C-12

APPENDIX C-1

ANALYSIS OF CALCIUM SILICATE (PORTLAND CEMENT) SORBENTS

OPERATION OF EPA OWNED TEST FACILITIES/TECHNICAL SUPPORT
--TECHNICAL SUPPORT OFFICE--

ANALYTICAL REPORT

RESULTS OF ANALYSES ON SAMPLES

ELEMENTAL ANALYSIS BY ICAP FOR LIMB

Prepared for:

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March 8, 1985

Prepared by:

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Contract No. 68-02-3988
Task No. 85-5
Report No. 7415.21-85-06

Approved By:

Acurex Chemistry Group Leader: ____________________ (L. Nelms)

Acurex QA Officer: ____________________ (S. Setzer)

INTRODUCTION

A sample described as treated portland cement was received from Jeff Lorrain for determination of metals by using Inductively Coupled Argon Plasma (ICAP) emission spectroscopy. These analyses were performed using the procedures developed by the Technical Support Office of the Air and Energy Engineering Research Laboratory (AEERL) of EPA. These procedures have been developed as a result of this sample set, so complete characterization of several parameters has not been completed at this time. However, in an effort to provide results in a timely fashion, this report was drafted subject to limitations as noted.

SAMPLE PREPARATION

As agreed, the sample was treated by different procedures for comparison purposes.

Procedure 1

The cement sample was treated as a limestone sample and subjected to a "total" digestion using a new procedure developed and implemented by Mr. David Natschke of Acurex, the onsite analytical services support contractor.

This sample preparation method required multi-step reactive dissolution of the cement, with neutralization of the reactive media after the sample was dissolved. First, a 0.1 gram portion of the sample was reacted with concentrated reagent grade nitric acid (HNO_3) at room temperature until all carbonate portions of the cement were dissolved. The mixture that resulted from this step was filtered through a Teflon membrane filter. The filtrate was retained, and any solids that remained on the filter were transferred to a Parr acid digestion bomb, where the residue was treated with reagent grade hydrofluoric acid at 140 degrees C. Any excess fluoride that remained after this step was complexed with boric acid prior to combining the fractions in order to prevent the precipitation of calcium ions from the solution.

Following digestion of the sample, both portions were combined and the volume adjusted to 100 mL. This solution contained the metal ions from the original cement, and was ready for analysis.

The method as described above is currently undergoing rigorous validation testing to determine limits of precision and accuracy. Upon completion of these tests, it will be written up and become the standard operating procedure (SOP) for the lab.

Procedure 2

The cement sample was treated with hydrochloric acid at room temperature until reaction ceased.

A 0.1 gram portion of the cement was weighed into a teflon liner and treated with minimal portions of reagent grade concentrated hydrochloric acid. When all signs of reaction ceased, deionized water was added and it was filtered through Whatman #41 paper which had been acid rinsed to reduce contamination. The filtrate was made up to 100 mL in a volumetric flask with deionized water.

Procedure 3

The cement sample was treated with refluxing hydrochloric acid.

A 0.1 gram sample was weighed into an erlenmeyer flask. A reflux cap was fitted to the flask, 5 mL of reagent grade concentrated hydrochloric acid was added, and the mixture was heated at a reflux for two hours on a hot plate. Deionized water was added and it was filtered through Whatman #41 filter paper which had been acid rinsed to reduce contamination. The filtrate was made up to 100 mL in a volumetric flask with deionized water.

Sample Analysis

All samples were analyzed for presence of certain elements using the standard ICAP file for multi-element analyses. First, the instrument was calibrated, using a range of standards which covered the working range of the technique. The solutions were aspirated into the plasma at a constant rate while the light that was emitted was scanned by the monochromator over the wavelength range for the various elemental emission lines. The intensities of these lines as specified by the data system were recorded and stored for generation of a calibration curve for each element. These calibration curves were used by the computer to calculate the concentration of each element found in each of the samples.

After the calibration curves were determined, each sample digest was aspirated into the plasma of the ICAP using the same conditions as for the standards. The response for each element was obtained and compared to that value for a reagent blank. If the response was greater than the blank, the blank correction was made, and a value for the element in the original sample, before it was dissolved was calculated. This value was compared to the available limits of detection, to determine if it was within the working range of the technique.

PRESENTATION OF RESULTS

The limits of detection for each element for which they are presently available is presented in Table 1. These reflect the fact that, at present, an insufficient number of samples has been run to provide accurate statistical values for these parameters. The data are, in fact, based on the estimated limits of detection in the standards, as specified by the instrument manufacturer. Since there are several factors that affect the data for the samples that are not a factor in the standards, these limits cannot yet provide a reliable estimate of overall precision and acuracy of the method.

The results of the sample analyses are presented in Tables 2 through 4. For each sample, those values that exceeded the estimated lower limit for quantification are given in weight percent in the original sample. The values that were below that limit are given as "BOL", meaning that detection was probable, but there was insufficient material present for reliable quantification. For any element that was lower than the detection limit cited in Table 1, the result is given as "BDL".

DISCUSSION

The sample analyses were performed with no experimental difficulites. The only significant difference between the results for the preparative procedures occurs for silicon using refluxed acid. This is not surprising as silicic acid tends to coagulate as silica gel under acidic conditions. This is accelerated by heat. The other elemental results for this preparative procedure tend to be lower. This may be explained in terms of losses due to occlusion within the silica gel. Coagulated silica gel may also be responsible for the slightly low calcium and silicon results when the sample was prepared using hydrofluoric acid. The teflon filter clogged badly and filtration occured over a long period (>24 hours). Errors of this magnitude could easily occur over this time period.

For these reasons, the results from treating the sample with hydrochloric acid at room temperature are probably the most dependable. All of the calcium results are in general agreement with those obtained previously by atomic absorption in the Fundamentals Laboratory. It should be noted that 35 percent calcium and 4 percent silicon does not agree with the mass balance for any ordinary calcium silicate. This material may be a mixture.

QA/AC

The data from elements with concentrations well above the estimated limits for quantification fall well within the guidelines for "good" QC.

Table 1

Element	Detection Limit as Weight %	Quantifiable Limit as Weight %
Ag	0.0002	0.001
Al	0.003	0.015
As	0.004	0.02
Ba	0.00004	0.0002
Be	0.00002	0.0001
Ca	0.003	0.015
Cd	0.0002	0.001
Co	0.0002	0.001
Cr	0.0002	0.001
Cu	0.0002	0.001
Fe	0.0002	0.001
Hg	0.001	0.005
K	0.01	0.05
Li	0.0003	0.0015
Mg	0.005	0.025
Mn	0.0001	0.0005
Na	0.002	0.01
Ni	0.0004	0.002
2316 P	0.002	0.01
Pb	0.004	0.02
S	0.002	0.01
Sb	0.004	0.02
Se	0.006	0.03
Si	0.002	0.01
Sn	0.003	0.015
Sr	TBD	TBD
Ti	0.0002	0.001
Tl	0.005	0.025
V	0.0005	0.0025
Zn	0.0001	0.0005

TBD = To Be Determined

Table 2. Treated Portland Cement
Digested W/HF

		Weight %
	Ca /	32.5
	Si /	4.04
	Al /	1.83
	Fe /	1.11
	Mg /	1.61
	K /	.103
	Mn /	.0482
	Na /	.114
2316	P /	.0577
	Ti /	.0893
	Ag /	BDL
	Ba /	.01
	Be /	.000173
	Cd /	BDL
	Co /	.00216
	Cr /	.00498
	Cu /	.00628
	Li /	BDL
	Ni /	.00814
	Pb /	BDL
	S /	.861
	Sb /	BDL
	Sn /	BDL
	Sr /	.156
	V /	BDL
	Zn /	.00606

BDL = At or below the limit of detection

BQL = At or below the limit of quantification

Table 3. Treated Portland Cement
Refluxed W/HCl

wt. (g) / vol.(mL) / conc. /	Weight %
Ca /	35.0
Si /	.101
Al /	2.06
Fe /	.994
Mg /	1.54
K /	.142
Mn /	.0434
Na /	.0801
2316 P /	.0635
Ti /	.1
Ag /	BDL
Ba /	.0109
Be /	.000196
Cd /	BDL
Co /	.00244
Cr /	.00205
Cu /	.00142
Li /	.00161
Ni /	BQL
Pb /	BDL
S /	.924
Sb /	BDL
Sn /	BDL
Sr /	.163
V /	BDL
Zn /	.00147

BDL = At or below the limit of detection

BQL = At or below the limit of quantification

Table 4. Treated Portland Cement
Digested W/HCl (room temp)

wt. (g) / vol. (mL) / conc. /	Weight %
Ca /	36.9
Si /	4.15
Al /	2.14
Fe /	1.02
Mg /	1.48
K /	.193
Mn /	.0441
Na /	.0876
2316 P /	.061
Ti /	.0976
Ag /	BDL
Ba /	.0116
Be /	.000171
Cd /	BDL
Co /	.00251
Cr /	.00294
Cu /	.00128
Li /	BDL
Ni /	BDL
Pb /	BDL
S /	.973
Sb /	BDL
Sn /	BDL
Sr /	.171
V /	BDL
Zn /	.00166

BDL = At or below the limit of detection

BQL = At or below the limit of quantification

```
TREATED PORTLAND CEMENT DIGESTED W/HCl(room temp.)
wt. (g.) /
vol. mL. /
   conc. /   Wt. %
--------------------
      Ca /   36.9
      Si /    4.15
      Al /    2.14
      Fe /    1.02
      Mg /    1.48
       K /     .193
      Mn /     .0441
      Na /     .0876
  2316 P /     .061
      Ti /     .0976
      Ag /   BDL
      Ba /     .0116
      Be /     .000171
      Cd /   BDL
      Co /     .00251
      Cr /     .00294
      Cu /     .00128
      Li /   BDL
      Ni /   BDL
      Pb /   BDL
       S /     .973
      Sb /   BDL
      Sn /   BDL
      Sr /     .171
       V /   BDL
      Zn /     .00166

  BDL = AT OR BELOW THE LIMIT OF DETECTION

  BOL = AT OR BELOW THE LIMIT OF QUANTIFICATION
```

APPENDIX C.2

ANALYSIS OF DEMONSTRATION SORBENTS

OPERATION OF EPA OWNED
TEST FACILITIES/TECHNICAL SUPPORT
--TECHNICAL SUPPORT OFFICE--

ANALYTICAL REPORT

RESULTS OF ANALYSES ON SAMPLES

ELEMENTAL ANALYSIS BY ICAP FOR LIMB

Prepared for:

Mr. Samuel Rakes
EPA

October 9, 1985

Prepared by:

Mr. David F. Natschke

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Contract No. 68-02-3988
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Report No. 7415.21-85-10

Approved By:

Acurex Chemistry Group Leader: ------------------------ (L. Nelms)

Acurex QA Officer: ------------------------ (S. Setzer)

INTRODUCTION

Eightteen coal samples were received from Mr. Samuel Rakes for treatment by Low Temperature Asher and determination of metals by use of Inductively Coupled Argon Plasma (ICAP) emission spectroscopy. These analyses were performed using the procedures developed by the Technical Support Office of the Air and Energy Engineering Research Laboratory (AEERL) of EPA. These procedures have been developed as a result of LIMB samples.

SAMPLE PREPARATION

Low Temperature Ashing

As specified by the project officer, these samples were pre-treated by Low Temperature Ashing. This procedure decomposes organics in a solid sample by reaction with singlet oxygen from an oxygen plasma. This procedure is considered to provide good elemental recovery since the reaction occurs at a relatively low temperature - ~140 degrees Centigrade - which reduces losses by evaporation.

This work was initially sub-contracted to the Research Triangle Institute. However, when they were unable to provide results in a timely manner, a portion of this work was done in N-wing; first by personnel of AEERL/TSO and later by personnel from Acurex, the on-site analytical services support contractor.

An aliquot of the resulting ash was digested by a procedure similar to that used in this laboratory for previous LIMB reports. A second portion has been submitted to Dr. Dean Smith of AEERL/EPA for possible physical analysis.

'Total' Digest

The inorganic ash resulting from the above procedure was digested in a manner similar to that used for limestone and sorbent samples. This sample preparation method required multi-step reactive dissolution of the ash, with neutralization of the reactive media after the sample was dissolved. First, a 0.1 gram portion of the sample was reacted with concentrated reagent grade nitric acid (HNO_3) at room temperature until all carbonate portions of the limestone were dissolved. The mixture that resulted from this step was filtered through a Teflon membrane filter. The filtrate was retained, and any solids that remained on the filter were transferred to a Parr acid digestion bomb, where the residue was treated with reagent grade hydrofluoric acid at 140 degrees C. Any excess fluoride that remained after this step was complexed with boric acid prior to combining the fractions in order to prevent the precipitation of calcium ions from the solution.

Following digestion of the sample, both portions were combined and the volume adjusted to 100 mL. This solution contained the metal ions from the original coal sample, and was remained that were probably carbon remaining from an incomplete low temperature ashing.

The method as described has been written as a first draft standard operating procedure (SOP) for the lab and is presently in the review process preparatory to approval

SAMPLE ANALYSIS

All samples were analyzed for presence of certain elements using the standard ICAP file for multi-element analyses. First, the instrument was calibrated, using a range of standards which covered the working range of the technique. The solutions were aspirated into the plasma at a constant rate while the light that was emitted was scanned by the monochromator over the wavelength range for the various elemental emission lines. The intensities of these lines as specified by the data system were recorded and stored for generation of a calibration curve for each element. These calibration curves were used by the computer to calculate the concentration of each element found in each of the samples.

After the calibration curves were determined, each sample digest was aspirated into the plasma of the ICAP using the same conditions as for the standards. The response for each element was obtained and compared to that value for a reagent blank. If the response was greater than the blank, the blank correction was made, and a value for the element in the original sample, before it was dissolved was calculated. This value was compared to the available limits of detection, to determine if it was within the working range of the technique.

PRESENTATION OF RESULTS

The limits of detection and quantitation used in this report are presented in Table 2. These values are based upon the standard deviations actually obtained from replicate analyses of these sample digests and reagent blank. These values have been calculated on the basis of the original coal using the assumptions of digesting 0.1 gram of ash from a typical coal with 10% ash. Table 1 presents the actual % ash found for these samples.

The results of the sample analyses are presented in Tables 3-6. These values are calculated on the basis of the original coal sample using the % ash correction factors found in Table 1. Use Table 1 also to transfer between the AEERL/TSO sample codes used in Tables 3-10 and the labels found on the original sample vials. Those values that exceeded the estimated lower limit for quantification are given in weight percent in the original sample. The values that were below that limit are given as "BQL", meaning that detection was probable, but there was insuficient material present for reliable quantification. For any element that was lower than the detection limit cited in Table 2, the result is given as "N. D.". The spread in data for these analyses reflects imprecision arising from all experimental parameters, as well as from inhomogeneiety within the sample.

Two ashed residues were digested in duplicate. Tables 7& 8 compare the analytical results. Two coal samples were low temperature ashed in duplicate. Tables 9& 10 compare the analytical results for the digests of these duplicates. Figures 1-30 present the graphics for the digests of two coal samples and a reagent blank in the vicinity of each analytical wavelength. The two samples used for these graphs were selected as extreme cases for as many wavelengths as possible.

DISCUSSION

No particular analytical problems were encountered during the analysis of these samples. The Low Temperature Ashing step is particularly time consuming. Ashing a single sample can take more than a week.

QA/QC

The data from elements with concentrations well above the estimated limits for quantification fall well within the guidelines for 'good' QC. However, as the values approach that limit, the spread in data indicated for the sample duplicates become larger. It should be noted that the % RSD's for replicate ashings of the same coal tend to be higher than those for replicate digests of a single ashing. This would indicate that homogeneiety of the coal is part of the problem.

Figures 1-30 may be used to identify possible spectral interferences. Thallium does show a potential interferent. This is tentatively identified as due to titanium. Titanium has lines at 1908.29 and at 1909.33 angstroms[1]. Additionally, the ratio of the two samples used at titanium matches their ratio at the interfering lines. No false positives are reported for thallium despite this. Beryllium has been deleted from the reported elements. The peaks in Figure 5 are poorly shaped; indicative of an unresolved interfering line. Examination of the tabulated data reveals a corrleation between the calculated iron and beryllium values.

REFERENCES

1. An Atlas of Spectral Interferences in ICP Spectroscopy, p.567, by Parsons, Forster, Anderson

TSO #	% ASH	ASH WT	*COAL W LIME	SAMPLE DESCRIPTOR
400A	11.9	0.1008	0.8471	UTAH D 3/8/85
400B	16.2	0.1003	0.6191	ILLINOIS 6 3/13/85
400C	11.5	0.1002	0.8713	W. KENTUCKY D 3/8/85
400D	13.3	0.1024	0.7699	MONT. D 3/8/85
D400D	12.4	0.1002	0.8081	DUP lo T ash MONT D
400E	10.4	0.1006	0.9673	INDIANA 3/13/85
400F	11.7	0.1003	0.8573	UTAH C 3/8/85
400G	11.6	0.1006	0.8672	UTAH A 2/15/85
400H	10.2	0.1001	0.9814	W. KENTUCKY A 2/15/85
400I	10	0.1004	1.004	W. KENTUCKY B 3/8/85
401A	12.2	0.1002	0.8213	MONT. A 2/15/85
401B	9.9	0.1004	1.0141	PITTS #8 A 3/8/85
401C	14.9	0.1006	0.6752	MONT. B 3/8/85
401D	10.4	0.1035	0.9952	UTAH B 3/8/85
401E	12.7	0.1002	0.789	PITTS 8 C 3/8/85
401F	13.8	0.101	0.7319	W. KENTUCKY C 3/8/85
401FD	13.8	0.1006	0.729	DUP DIGEST lo T ashed W. KENTUCKY C
401G	11.9	0.1006	0.8454	MONT. C 3/8/85
D401G	12.1	0.1011	0.8355	DUP lo T ash of MONT C
401H	12.6	0.1025	0.8135	PITTS 8 B 3/8/85
401HD	12.6	0.1009	0.8008	DUP DIGEST lo T ashed PITTS 8 B
401I	12.1	0.1006	0.8314	PITTS 8 D 3/8/85

TABLE 1. SAMPLE DESCRIPTORS vs. TSO #

Note: 4 samples of each coal type were submitted.

Utah	Pitts 8	Montana
400A - D 3/08/85	401B - A 3/08/85	400D - D 3/08/85
400F - C 3/08/85	401E - C 3/08/85	401A - A 2/15/85
400G - A 2/15/85	401H - B 3/08/85	401C - B 3/08/85
401D - B 3/08/85	401I - D 3/08/85	401G - C 3/08/85

	STD DEV	DETECTION LIMIT WEIGHT %	QUANTIFIABLE LIMIT WEIGHT %
Ag	0.005	0.00015	0.00075
Al	0.057	0.00171	0.00855
As	0.036	0.00108	0.0054
Ba	0.004	0.00012	0.0006
Be	0.001	0.00003	0.00015
Ca	0.034	0.00102	0.0051
Cd	0.001	0.00003	0.00015
Co	0.005	0.00015	0.00075
Cr	0.006	0.00018	0.0009
Cu	0.005	0.00015	0.00075
Fe	0.006	0.00018	0.0009
Hg	0.03	0.0009	0.0045
K	0.24	0.0072	0.036
Li	0.003	0.00009	0.00045
Mg	0.154	0.00462	0.0231
Mn	0.003	0.00009	0.00045
Na	0.038	0.00114	0.0057
Ni	0.003	0.00009	0.00045
P	0.042	0.00126	0.0063
Pb	0.1	0.003	0.015
S	0.148	0.00444	0.0222
Sb	0.097	0.00291	0.01455
Se	1.523	0.04569	0.22845
Si	0.059	0.00177	0.00885
Sn	0.025	0.00075	0.00375
Sr	0.001	0.00003	0.00015
Ti	0.006	0.00018	0.0009
Tl	0.3	0.009	0.045
V	0.002	0.00006	0.0003
Zn	0.002	0.00006	0.0003

Table 2. Detection & Quantifiable Limits for Coal

	Utah 400A	400B	400C	Mont. 400D	400E
	Wt. %	Wt. %	Wt. %	Wt. %	Wt. %
Ag	N. D.	N. D.	N. D.	N. D.	N. D.
✓Al	.764	.798	.287	.589	.316
As	N. D.	BQL	N. D.	N. D.	BQL
Ba	.00299	.00237	BQL	.0108	.000951
Be					
✓Ca	.259	.366	.0289	.938	.0263
Cd	.000165	.000291	BQL	N. D.	.000222
Co	.000944	.00136	.00118	.000805	.0018
✓Cr✓	.00272	.00226	.00127	.00225	.00124
Cu	BQL	BQL	BQL	BQL	.00188
Fe	.255	1.17	1.25	.217	.967
Hg	BQL	N. D.	N. D.	N. D.	N. D.
✓K ✓	.0648	.119	.0791	.0361	.0647
Li	.00138	.000485	BQL	.00126	.000806
✓Mg	.0666	.0444	BQL	.291	BQL
Mn	.00123	.00455	.00135	.0103	.00117
✓Na✓	.0282	.0427	.00846	.0198	.0103
Ni	.000933	.00147	N. D.	.000649	.00527
P	.0147	.00716	BQL	.00856	.00724
Pb	BQL	BQL	N. D.	N. D.	BQL
S	.475	1.84	1.4	.531	.843
Sb	N. D.	N. D.	N. D.	N. D.	N. D.
Se	N. D.	BQL	N. D.	N. D.	BQL
Si	3.03	2.37	1.43	1.93	1.17
Sn	N. D.	N. D.	N. D.	N. D.	N. D.
Sr	.00994	.00228	.000677	.0325	.00247
Ti	.0661	.0598	.0491	.0556	.0383
Tl	N. D.	N. D.	N. D.	N. D.	N. D.
V	.00112	.00202	.0027	.000662	.00152
Zn	.0012	.00515	.00398	.000546	.0121

Table 3. Analytical Results for Coal Samples.

N. D.= not detected
BQL = below the quantifiable limit

	Utah	Utah			Mont.
	400F	400G	400H	400I	401A
	Wt. %	Wt. %	Wt. %	Wt. %	Wt. %
Ag	N. D.	N. D.	N. D.	BQL	N. D.
✓ Al	.049	.433	.319	.489	.567
As	N. D.	N. D.	N. D.	N. D.	N. D.
Ba	.00439	.00152	.000846	.000901	.0111
Be					
✓ Ca	.274	.239	.0407	.0719	.046
Cd	.000175	N. D.	.000173	.000463	N. D.
Co	.000956	N. D.	.00118	.0029	N. D.
✓ Cr	.0116	.00316	.00105	.00153	.00324
Cu	.00084	BQL	BQL	BQL	BQL
Fe	.225	.227	1.2	1.24	.223
Hg	N. D.	N. D.	N. D.	BQL	N. D.
✓ K	.072	.0448	.0807	.0661	BQL (.0360)
Li	.00135	.00108	BQL	BQL	.0011
✓ Mg	.0771	.0607	BQL	.0235	.276
Mn	.00098	.001	.00141	.00302	.00948
✓ Na	.0291	.0253	.00762	.00936	.0186
Ni	.00119	N. D.	N. D.	.000986	N. D.
P	.0148	.013	BQL	.00759	.00776
Pb	BQL	N. D.	N. D.	BQL	N. D.
S	.454	.51	.969	.711	.6
Sb	N. D.	N. D.	N. D.	N. D.	N. D.
Se	N. D.	N. D.	N. D.	N. D.	N. D.
Si	2.63	2.78	1.47	1.51	1.83
Sn	N. D.	N. D.	N. D.	N. D.	N. D.
Sr	.0107	.00773	.000958	.00149	.031
Ti	.0637	.0648	.0466	.0488	.0478
Tl	N. D.	N. D.	N. D.	N. D.	N. D.
V	.00106	.000726	.00265	.00275	.000341
Zn	.00104	.000819	.00352	.0032	.000335

Table 4. Analytical Results for Coal Samples.

N. D.= not detected
BQL = below the quantifiable limit

	Pitt8	Mnt	Utah	Pitt 8	
sam	401B	401C	401D	401E	401F
wt.					
vol					
con	Wt. %	Wt. %	Wt. %	Wt. %	Wt. %
Ag	N. D.	N. D.	N. D.	N. D.	N. D.
✓ Al	.721	.661	.671	.684	.659
As	N. D.	N. D.	N. D.	BQL	N. D.
Ba	.0094	.0218	.00279	.00812	.000696
Be					
✓ Ca	.434	1.08	.249	.412	.0514
Cd	.000167	N. D.	BQL	.000234	.00043
Co	.000937	.0098	BQL	.00137	.00318
✓ Cr	.00232	BQL (.0009)	.00217	.00151	.00208
Cu	BQL	BQL	BQL	BQL	.000674
Fe	.766	.282	.214	1.25	1.81
Hg	N. D.	N. D.	BQL	N. D.	BQL
✓ K	.0669	BQL (.0360)	.0589	.0787	.123
Li	.000661	.00151	.00121	.00064	.00056
✓ Mg	.0741	.304	.0659	.0667	.027
Mn	.0024	.0118	.0011	.00265	.00212
✓ Na	.0449	.0234	.0266	.0415	.0105
Ni	N. D.	.000459	.000965	.000684	.00238
P	.0136	.0092	.0138	.0162	.00741
Pb	N. D.	BQL	BQL	BQL	BQL
S	.994	.636	.469	1.67	1.5
Sb	N. D.	N. D.	N. D.	N. D.	N. D.
Se	N. D.	N. D.	N. D.	BQL	N. D.
Si	1.51	2.32	2.54	1.69	2.08
Sn	N. D.	N. D.	N. D.	N. D.	N. D.
Sr	.0124	.0367	.00916	.011	.000896
Ti	.0476	.0637	.0612	.0547	.0673
Tl	N. D.	N. D.	N. D.	N. D.	N. D.
V	.00107	.000481	.000985	.00118	.00414
Zn	.000695	.000348	.00103	.000925	.00555

Table 5. Analytical Results for Coal Samples.

N. D.= not detected
BQL = below the quantifiable limit

	mat 401G Wt. %	P.H 8 401H Wt. %	P.H 8 401I Wt. %
Ag	N. D.	N. D.	N. D.
Al	.487	.467	.409
As	N. D.	N. D.	BQL
Ba	.0191	.00793	.00707
Be			
Ca	.893	.445	.393
Cd	N. D.	.000166	.000217
Co	N. D.	.00199	.00146
Cr	.00856	.00124	.00126
Cu	BQL	BQL	.000872
Fe	.356	1.34	1.23
Hg	N. D.	N. D.	N. D.
K	BQL (.036)	.0616	.0582
Li	.00102	.000639	.000595
Mg	.263	.0317	.0483
Mn	.0101	.00268	.00266
Na	.0172	.0514	.0427
Ni	N. D.	.00124	.000674
P	.00713	.0165	.0155
Pb	N. D.	N. D.	BQL
S	.859	.996	1.23
Sb	N. D.	N. D.	N. D.
Se	N. D.	N. D.	N. D.
Si	1.73	1.86	1.65
Sn	N. D.	N. D.	N. D.
Sr	.03	.01	.00919
Ti	.0456	.0602	.0512
Tl	N. D.	N. D.	N. D.
V	N. D.	.00122	.00112
Zn	.000408	.000977	.00074

Table 6. Analytical Results for Coal Samples.

N. D.= not detected
BQL = below the quantifiable limit

	401H	401HD			
	Wt. %	Wt. %	AVERAGE	std dev	% RSD
Ag	N. D.	N. D.			
Al	.467	.401	.474	0.01059	2
As	N. D.	N. D.			
Ba	.00793	.00817	.00805	0.00017	2
Be					
Ca	.445	.443	.444	0.00148	0
Cd	.000166	.000212	.000189	0.00003	17
Co	.00199	.00206	.00203	0.00005	2
Cr	.00124	.00128	.00126	0.00003	3
Cu	BQL	BQL			
Fe	1.34	1.35	1.34	0.01228	1
Hg	N. D.	N. D.			
K	.0616	.0702	.0659	0.00610	9
Li	.000639	.000687	.000663	0.00003	5
Mg	.0517	.0604	.056	0.00618	11
Mn	.00268	.00277	.00273	0.00007	2
Na	.0514	.0488	.0501	0.00185	4
Ni	.00124	.00115	.00119	0.00006	5
P	.0165	.017	.0168	0.00041	2
Pb	N. D.	BQL			
S	.996	.998	.997	0.00107	0
Sb	N. D.	N. D.			
Se	N. D.	N. D.			
Si	1.86	1.92	1.89	0.04114	2
Sn	N. D.	N. D.			
Sr	.01	.0107	.0104	0.00049	5
Ti	.0602	.0602	.0602	0.00001	0
Tl	N. D.	N. D.			
V	.00122	.00127	.00125	0.00004	3
Zn	.000977	.000824	.000901	0.000108	12

Table 7. Comparison of Duplicate Digestions

N. D.= not detected
BQL= below the quantifiable limit

	401F Wt. %	401FD Wt. %	AVERAGE	Std dev	% RSD
Ag	N. D.	N. D.			
Al	.659	.553	.606	0.07501	12
As	N. D.	N. D.			
Ba	.000695	.000809	.000652	0.00006	7
Be					
Ca	.0514	.103	.077	0.03623	47
Cd	.00043	.000302	.000366	0.00009	25
Co	.00318	.00258	.00288	0.00043	15
Cr	.00207	.00212	.00209	0.00003	2
Cu	.000874	BQL			
Fe	1.81	1.73	1.77	0.05488	3
Hg	BQL	N. D.			
K	.123	.11	.116	0.00924	8
Li	.00056	BQL			
Mg	.027	.0236	.0253	0.00238	9
Mn	.00212	.00218	.00215	0.00004	2
Na	.0105	.0114	.011	0.00066	6
Ni	.00238	.00181	.0021	0.00041	19
P	.00741	BQL			
Pb	BQL	N. D.			
S	1.5	1.45	1.48	0.02993	2
Sb	N. D.	N. D.			
Se	N. D.	N. D.			
Si	2.08	2	2.04	0.05845	3
Sn	N. D.	N. D.			
Sr	.000895	.001	.000948	0.00008	8
Ti	.0673	.0664	.0668	0.00069	1
Tl	N. D.	N. D.			
V	.00414	.00373	.00394	0.00029	7
Zn	.00555	.00551	.00553	0.00002	0

Table 8. Comparison of Duplicate Digests

N. D.= not detected
BQL= below the quantifiable limit

	400D	D400D			
	Wt. %	Wt. %	AVERAGE	std dev	% RSD
Ag	N. D.	N. D.			
Al	.589	.743	.666	0.10893	16
As	N. D.	N. D.			
Ba	.0108	.0135	.0122	0.00192	16
Be	.00326	.00325	.00326	0.00000	0
Ca	.938	.866	.902	0.05070	6
Cd	N. D.	.000223			
Co	.000805	.00106	.000935	0.00018	20
Cr	.00225	.0156	.00894	0.00946	106
Cu	BQL	.001			
Fe	.217	.219	.218	0.00122	1
Hg	N. D.	BQL			
K	.0361	.0422	.0391	0.00430	11
Li	.00126	.00113	.00119	0.00009	8
Mg	.291	.267	.279	0.01700	6
Mn	.0103	.00976	.01	0.00037	4
Na	.0198	.0213	.0206	0.00107	5
Ni	.000649	.000879	.000764	0.00016	21
P	.00856	.00877	.00867	0.00015	2
Pb	N. D.	N. D.			
S	.531	.545	.538	0.00994	2
Sb	N. D.	N. D.			
Se	N. D.	N. D.			
Si	1.93	1.74	1.84	0.13817	8
Sn	N. D.	N. D.			
Sr	.0325	.0312	.0318	0.00087	3
Ti	.0556	.0502	.0529	0.00385	7
Tl	N. D.	N. D.			
V	.000662	.000594	.000628	0.00005	8
Zn	.000546	.000433	.000489	0.00008	16

Table 9. Comparison of Duplicate LoTemp Ashings

N. D.= not detected
BQL= below the quantifiable limit

	401G	D401G			
	Wt. %	Wt. %	AVERAGE	std dev	% RSD
Ag	N. D.	N. D.			
Al	.487	.706	.596	0.15529	26
As	N. D.	N. D.			
Ba	.0191	.00886	.014	0.00726	52
Be					
Ca	.893	.955	.924	0.04380	5
Cd	N. D.	.000443			
Co	N. D.	.00218			
Cr	.00855	.00101	.00478	0.00534	112
Cu	BQL	BQL			
Fe	.356	.403	.38	0.03328	9
Hg	N. D.	BQL			
K	BQL	.0484			
Li	.00102	.000999	.00101	0.00001	1
Mg	.265	.241	.253	0.01695	7
Mn	.0101	.0135	.0118	0.00236	20
Na	.0172	.0173	.0173	0.00006	0
Ni	N. D.	N. D.			
P	.00713	.0116	.00937	0.00316	34
Pb	N. D.	BQL			
S	.859	.559	.709	0.21181	30
Sb	N. D.	N. D.			
Se	N. D.	N. D.			
Si	1.73	1.94	1.83	0.14321	8
Sn	N. D.	N. D.			
Sr	.03	.0321	.0311	0.00146	5
Ti	.0456	.0491	.0473	0.00254	5
Tl	N. D.	N. D.			
V	N. D.	.000371			
Zn	.000408	.000527	.000467	0.00008	18

Table 10. Comparison of Duplicate LoTemp Ashings

N. D.= not detected
BQL= below the quantifiable limit

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  AG 3260.66   ***             27-SEP-65



STEP  ANG.       INCREMENT =  421.1 COUNTS     FULL SCALE = 25268. COUNTS

              I....I....I....I....I....I....I....I....I....I....I....I....I
-11  3260.06                                                        .*C
-10  3260.12                                                       . C
-9   3260.17                                                        .*C
-8   3260.23                                                        .*C
-7   3260.29                                                        *.
-6   3260.34                                                        .C
-5   3260.40                                                       .*C
-4   3260.45                                                       .C
-3   3260.51                                                        .C
-2   3260.57                                                        .*
-1   3260.62                                                       C.
 0   3260.66                                                       C.
 1   3260.74                                                      .C*
 2   3260.79                                                       .C
 3   3260.85                                                        .
 4   3260.90                                                        .C
 5   3260.96                                                        C.
 6   3261.02                                                         .
 7   3261.07                                                          .
 8   3261.13                                                           .C
 9   3261.19                                                            .C
10   3261.24                                                             .C
11   3261.30                                                             .C
              I....I....I....I....I....I..............I....I....I....I....I
```

* = TOTAL DIGEST OF LTA'D COAL 401G

C = COAL SAMPLE 400C

. = REAGENT BLANK FOR COAL DIGESTS

Figure 1

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  AL 3062.15   ***                 07-SEP-85


STEP  ANG.        INCREMENT =  661.9 COUNTS     FULL SCALE = 40912. COUNTS

              I....I....I....I....I....I....I....I....I....I....I....I....I
-11  3061.53                 .*O
-10  3061.59                   .
-9   3061.64                 O.
-8   3061.70              O*.
-7   3061.76            O.
-6   3061.81           .
-5   3061.87          .O
-4   3061.92          .  O*
-3   3061.98          .      O  *
-2   3062.04           .              O      *
-1   3062.09           .                         O            *
 0   3062.15           .                               O                  *
 1   3062.21          .                        O                  *
 2   3062.26          .          O            *
 3   3062.32          .  O    *
 4   3062.37          .O*
 5   3062.43          .*
 6   3062.49          .
 7   3062.54          .
 8   3062.60
 9   3062.66          .
 10  3062.71          .
 11  3062.77          .
              I....I....I....I....I....I....I....I....I....I....I....I....I


      * = TOTAL DIGEST OF LTA'D COAL 401G

      O = COAL SAMPLE 400C

      . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 2

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  AS 1972.62   ***               27-SEP-85



STEP  ANG.        INCREMENT =   47.3 COUNTS      FULL SCALE = 2837.0 COUNTS

                 I....I....I....I....I....I....I....I....I....I....I....I....I
-11  1972.00                                                        G *.
-10  1972.06                                                         * .
 -9  1972.11                                                          .G
 -8  1972.17                                                           . G
 -7  1972.23                                                          *.
 -6  1972.28                                                         * .  G
 -5  1972.34                                                             .
 -4  1972.39                                                          .  *
 -3  1972.45                                                          .
 -2  1972.51                                                      G .
 -1  1972.56                                                      . G
  0  1972.62                                                        .* G
  1  1972.68                                                         .G
  2  1972.73                                                          . *
  3  1972.79                                                          . *G
  4  1972.84                                                            .
  5  1972.90                                                            G  .
  6  1972.96                                                              *.
  7  1973.01                                                                G.
  8  1973.07                                                             *. G
  9  1973.13                                                            .   G
 10  1973.18                                                          G .
 11  1973.24                                                         .G
                 I....I....I....I....I....I....I....I....I....I....I....I....I


            * = TOTAL DIGEST OF LTA'D COAL 401G

            G = COAL SAMPLE 400C

            . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 3

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  BA 4554.03   ***                     27-SEP-85


STEP  ANG.        INCREMENT = 1123.2 COUNTS      FULL SCALE = 67392. COUNTS

               I....I....I....I....I....I....I....I....I....I....I....I....I
-11  4553.41        .
-10  4553.47        .
 -9  4553.52        .
 -8  4553.58        .*
 -7  4553.64        .*
 -6  4553.69        .*
 -5  4553.75        .*
 -4  4553.80        .  *
 -3  4553.86        .     *
 -2  4553.92        .O            *
 -1  4553.97        .O                                 *
  0  4554.03        . O                                                  *
  1  4554.09        . O                                                    *
  2  4554.14        .O                                   *
  3  4554.20        .O                *
  4  4554.25        .    *
  5  4554.31        . *
  6  4554.37        .*
  7  4554.42        .*
  8  4554.48        .*
  9  4554.54        .*
 10  4554.59        .
 11  4554.65        .
               I....I....I....I....I....I....I....I....I....I....I....I....I


      * = TOTAL DIGEST OF LTA'D COAL 401O

      O = COAL SAMPLE 400C

      . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 4

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  BE 2348.61   ***                  27-SEP-83


STEP  ANG.      INCREMENT = 1441.5 COUNTS     FULL SCALE = 86512. COUNTS

               I....I....I....I....I....I....I....I....I....I....I....I....I
-11  2347.99      .
-10  2348.05      .
 -9  2348.10      .O
 -8  2348.16       .O
 -7  2348.22       .*O
 -6  2348.27      .  *    O
 -5  2348.33      .      *              O
 -4  2348.38      .         *                             O
 -3  2348.44      .           *                                        O
 -2  2348.50      .             *                                      O
 -1  2348.55      .              *                                      O
  0  2348.61     .                *                                        O
  1  2348.67     .           *                                    O
  2  2348.72      .    *                       O
  3  2348.78      .*       O
  4  2348.83      . O
  5  2348.89      . O
  6  2348.95      .O
  7  2349.00      .O
  8  2349.06      .O
  9  2349.12       .
 10  2349.17       .
 11  2349.23      *.
               I....I....I....I....I....I....I....I....I....I....I....I....I


      * = TOTAL DIGEST OF LTA'D COAL 401B

      O = COAL SAMPLE 400C

      . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 5

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  CA 4226.73   ***             27-SEP-85



STEP  ANG.        INCREMENT =   47.5 COUNTS      FULL SCALE = 2848.0 COUNTS

                I....I....I....I....I....I....I....I....I....I....I....I....I
 -11  4226.11      .
 -10  4226.17      .
  -9  4226.22      .
  -8  4226.28      .*
  -7  4226.34      .*
  -6  4226.39      .*
  -5  4226.45      . *
  -4  4226.50      .   *
  -3  4226.56      .       *
  -2  4226.62      .O                 *
  -1  4226.67      .O                                     *
   0  4226.73      . O                                                    *
   1  4226.79      . O                                                      *
   2  4226.84      . O                                *
   3  4226.90      .O          *
   4  4226.95      .  *
   5  4227.01      .*
   6  4227.07      .*
   7  4227.12      .*
   8  4227.18      .*
   9  4227.24      .*
  10  4227.29      .
  11  4227.35      .
                I....I....I....I....I....I....I....I....I....I....I....I....I


     * = TOTAL DIGEST OF LTA'D COAL 4016

     O = COAL SAMPLE 4000

     . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 6

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  CD 2144.36                    27-SEP-85


STEP  ANG.       INCREMENT =  156.0 COUNTS     FULL SCALE = 9360.0 COUNTS

              I....I....I....I....I....I....I....I....I....I....I....I....I
-11  2143.76                                                        .*
-10  2143.82                                                           .G
-9   2143.87                                                             .*
-8   2143.93                                                           .G
-7   2143.99                                                       *G.
-6   2144.04                                                 .
-5   2144.10                                            .G
-4   2144.15                                            .G
-3   2144.21                                                .*
-2   2144.27                                                    .*
-1   2144.32                                                    * .
 0   2144.38                                                    . G
 1   2144.44                                                    .* .
 2   2144.49                                                    . G
 3   2144.55                                                     .G
 4   2144.60                                                       .G*
 5   2144.66                                                         .G*
 6   2144.72                                                          .G
 7   2144.77                                                       .G
 8   2144.83                                                 *.G
 9   2144.88                                              .
10   2144.94                                         . G*
11   2145.00                                           . G*
              I....I....I....I....I....I....I....I....I....I....I....I....I

      * = TOTAL DIGEST OF LTA'D COAL 401G

      G = COAL SAMPLE 400C

      . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 7

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  CO 2376.62    ***                    27-SEP-85



STEP  ANG.       INCREMENT = 1932.3 COUNTS     FULL SCALE = 115938 COUNTS

              I....I....I....I....I....I....I....I....I....I....I....I....I
-11  2376.00              .C
-10  2376.06              .C
-9   2376.11              .C
-8   2376.17              .
-7   2376.23              .
-6   2376.28              .C
-5   2376.34              .C
-4   2376.39              . C
-3   2376.45              . C
-2   2376.51              . C
-1   2376.56              . C
 0   2376.62              .*C
 1   2376.68              .*C
 2   2376.73              .C
 3   2376.79              .C
 4   2376.84              .C
 5   2376.90              .C
 6   2376.96              .C
 7   2377.01              .*C
 8   2377.07              .*  C
 9   2377.13              . *     C
 10  2377.18              .     *             C
 11  2377.24              .             *                                 C
              I....I....I....I....I....I....I....I....I....I....I....I....I


     * = TOTAL DIGEST OF LTA'D COAL 4016

     C = COAL SAMPLE 4002

     . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 8

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  CR 2055.52    ***            27-SEP-85


STEP  ANG.       INCREMENT =  121.3 COUNTS     FULL SCALE = 7278.0 COUNTS

               I....I....I....I....I....I....I....I....I....I....I....I....I
-11  2054.90                               . O*
-10  2054.96                               .
 -9  2055.01                             . O
 -8  2055.07                            .  O
 -7  2055.13                              .O
 -6  2055.18                              .* O
 -5  2055.24                              .*  O
 -4  2055.29                              .  O
 -3  2055.35                              . O*
 -2  2055.41                              .  O   *
 -1  2055.46                              .   O            *
  0  2055.52                               .   O                        *
  1  2055.58                               .     O                          *
  2  2055.63                              .    O                *
  3  2055.69                               .  O      *
  4  2055.74                                .O
  5  2055.80                                .
  6  2055.86                               .
  7  2055.91                              .O
  8  2055.97                             .*
  9  2056.03                             O.
 10  2056.08                             .
 11  2056.14                           . O
               I....I....I....I....I....I....I....I....I....I....I....I....I


     * = TOTAL DIGEST OF LTA'D COAL 401G

     O = COAL SAMPLE 400C

     . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 9

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  CU 3247.54   ***               27-SEP-85


STEP  ANG.        INCREMENT =  388.7 COUNTS     FULL SCALE = 23324. COUNTS

              I....I....I....I....I....I....I....I....I....I....I....I....I
-11  3246.92                                                      .O
-10  3246.98
 -9  3247.03                                                      .O
 -8  3247.09                                                      .O
 -7  3247.15                                                       .O
 -6  3247.20                                                        .O
 -5  3247.26                                                         .O
 -4  3247.31                                                         .O
 -3  3247.37                                                         .O
 -2  3247.43                                                        .O
 -1  3247.48                                                        . O
  0  3247.54                                                         .  O*
  1  3247.60                                                         .  O *
  2  3247.65                                                          .  O*
  3  3247.71                                                          .O*
  4  3247.76                                  *                 .*
  5  3247.82                                                       .O
  6  3247.88                                                       .O
  7  3247.93                                                       .O
  8  3247.99                                                       .O
  9  3248.05                                                      O.*
 10  3248.10                                                      .O
 11  3248.16                                                      .O
              I....I....I....I....I....I....I....I....I....I....I....I....I


     * = TOTAL DIGEST OF LTA'D COAL 401G

     O = COAL SAMPLE 400C

     . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 10

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  FE 2599.4   ***              27-SEP-85



STEP  ANG.       INCREMENT = 1866.7 COUNTS     FULL SCALE = 113200 COUNTS

                I....I....I....I....I....I....I....I....I....I....I....I....I
-11  2598.78    .O
-10  2598.84    .O
-9   2598.89    .O
-8   2598.95    .O
-7   2599.01    .O
-6   2599.06    .O
-5   2599.12    .O
-4   2599.17    .* O
-3   2599.23    . *    O
-2   2599.29    .    *              O
-1   2599.34    .        *                                 O
 0   2599.40    .             *                                             O
 1   2599.46    .             *                                    O
 2   2599.51    .        *                 O
 3   2599.57    .  *     O
 4   2599.62    .*O
 5   2599.68    .O
 6   2599.74    .O
 7   2599.79    .O
 8   2599.85    .
 9   2599.91    .
 10  2599.96    .
 11  2600.02    .
                I....I....I....I....I....I....I....I....I....I....I....I....I


      * = TOTAL DIGEST OF LTA'D COAL 4010

      O = COAL SAMPLE 4000

      . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 11

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES*** HG 1942.27   ***                  27-SEP-85


STEP  ANG.       INCREMENT =   45.3 COUNTS     FULL SCALE = 2720.0 COUNTS

              I....I.........I....I....I....I....I....I....I....I....I....I
-11  1941.65                                                     .C
-10  1941.71                                                         .C
-9   1941.76                                                       . *
-8   1941.82                                                        .C
-7   1941.88                                                      *C.
-6   1941.93                                                      .C
-5   1941.99                                                    C.
-4   1942.04                                                      *. C
-3   1942.10                                                       .C
-2   1942.16                                                        C .
-1   1942.21                                                        .C
 0   1942.27                                                       C  .
 1   1942.33                                                         .*
 2   1942.38                                                       .
 3   1942.44                                                        .
 4   1942.49                                                       . C
 5   1942.55                                                      C *.
 6   1942.61                                                     *.
 7   1942.66                                                  *C  .
 8   1942.72                                                    . C*
 9   1942.78                                                        .*
 10  1942.83                                                          C .
 11  1942.89                                                            .C*
              I....I....I....I....I....I....I....I....I....I....I....I....I


      * = TOTAL DIGEST OF LTA'D COAL 401G

      C = COAL SAMPLE 400C

      . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 12

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  K 7664.91   ***               27-SEP-85


STEP ANG.        INCREMENT =   40.5 COUNTS     FULL SCALE = 2430.0 COUNTS

              I....I....I....I....I....I....I....I....I....I....I....I....I
-11  7664.29                                      .* O
-10  7664.35                                       *.
-9   7664.40                                       *.O
-8   7664.46                                       .
-7   7664.52                                      O.
-6   7664.57                                       .*
-5   7664.63                                      .O*
-4   7664.68                                        .O
-3   7664.74                                       .O
-2   7664.80                                        .  O
-1   7664.85                                        .*        O
 0   7664.91                                        .  *            O
 1   7664.97                                        .     *               O
 2   7665.02                                        .    *            O
 3   7665.08                                      .  *      O
 4   7665.13                                       * .O
 5   7665.19                                       *.
 6   7665.25                                       .*
 7   7665.30                                       . *
 8   7665.36                                        .
 9   7665.42                                       *.
 10  7665.47                                       .O
 11  7665.53                                        .
              I....I....I....I....I....I....I....I....I....I....I....I....I


      * = TOTAL DIGEST OF LTA'D COAL 4019

      O = COAL SAMPLE 4009

      . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 13

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  LI 6707.8   ***              27-SEP-85


STEP  ANG.        INCREMENT =  108.0 COUNTS      FULL SCALE = 6479.0 COUNTS

              I....I....I....I....I....I....I....I....I....I....I....I....I
-11  6707.18                                                .O
-10  6707.24                                                 .*
 -9  6707.29                                                  .
 -8  6707.35                                                 .
 -7  6707.41                                                 O.
 -6  6707.46                                                O*.
 -5  6707.52                                                 .*
 -4  6707.57                                                 . *
 -3  6707.63                                                  .O *
 -2  6707.69                                                 .  O   *
 -1  6707.74                                                 .   O      *
  0  6707.80                                                  .   O       *
  1  6707.85                                                 .    O     *
  2  6707.91                                                .    O      *
  3  6707.97                                                 . O     *
  4  6708.02                                                 . O   *
  5  6708.08                                                   .
  6  6708.14                                                 O.
  7  6708.19                                                . *
  8  6708.25                                                .O *
  9  6708.31                                                 .
 10  6708.36                                                 .O
 11  6708.42                                                *.
              I....I....I....I....I....I....I....I....I....I....I....I....I


     * = TOTAL DIGEST OF LTA'D COAL 401G

     O = COAL SAMPLE 400C

     . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 14

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES*** MG 2750.8   ***                 27-SEP-85


STEP  ANG.       INCREMENT =   60.1 COUNTS     FULL SCALE = 3604.0 COUNTS

              I....I....I....I....I....I....I....I....I....I....I....I....I
-11  2750.18                   .*
-10  2750.24                   O.
 -9  2750.29                   .*
 -8  2750.35                   .*
 -7  2750.41                   . *
 -6  2750.46                    .*
 -5  2750.52                    .*
 -4  2750.57                    . *
 -3  2750.63                    .  *
 -2  2750.69                   .O       *
 -1  2750.74                   . O                   *
  0  2750.80                   . O                                  *
  1  2750.86                   . O                                        *
  2  2750.91                   . O                             *
  3  2750.97                   .O              *
  4  2751.02                   .O   *
  5  2751.08                   . *
  6  2751.14                   . *
  7  2751.19                   .*
  8  2751.25                   .*
  9  2751.31                   .*
 10  2751.36                   . *
 11  2751.42                   . *
              I....I....I....I....I....I....I....I....I....I....I....I....I


    * = TOTAL DIGEST OF LTA'D COAL 401G

    O = COAL SAMPLE 400C

    . = REAGENT BLANK FOR COAL DIGESTS
```

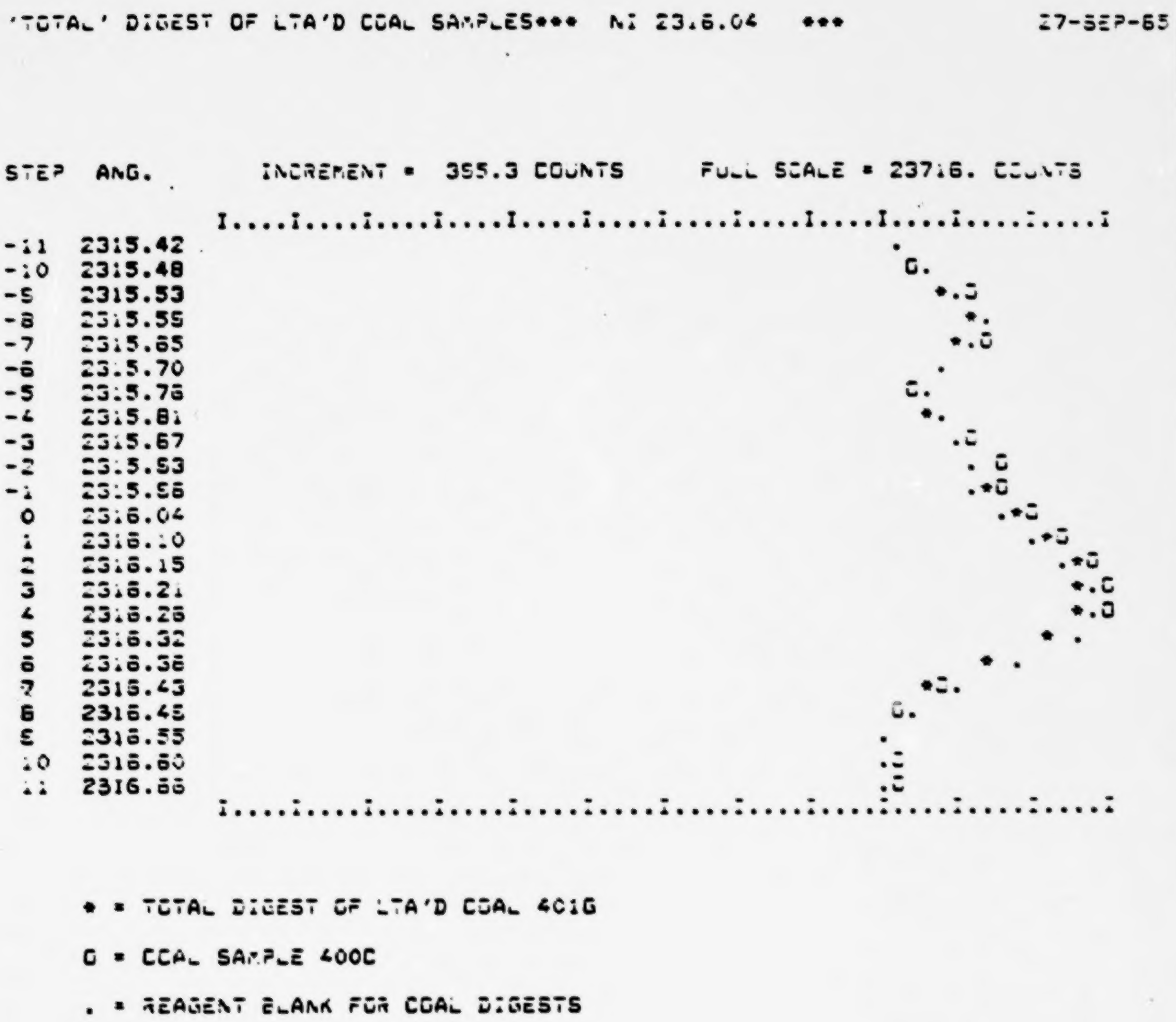
Figure 15

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  MN 2576.1   ***                    27-SEP-83


STEP  ANG.        INCREMENT =   62.6 COUNTS     FULL SCALE = 3756.0 COUNTS

              I....I....I....I....I....I....I....I....I....I....I....I....I
-11  2575.46             . O
-10  2575.54             .O
 -9  2575.59             .*
 -8  2575.65             .O
 -7  2575.71             .O
 -6  2575.76             .O
 -5  2575.82             .*O
 -4  2575.87             .*O
 -3  2575.93             .O  *
 -2  2575.98             . O       *
 -1  2576.04              .  O                     *
  0  2576.10              .     O                                 *
  1  2576.16              .      O                                        *
  2  2576.21              .     O                                *
  3  2576.27              .  O                 *
  4  2576.32             .O   *
  5  2576.38             .O
  6  2576.44             .
  7  2576.49             .
  8  2576.55            *.
  9  2576.61            .O
 10  2576.66            *.O
 11  2576.72            .*O
              I....I....I....I....I....I....I....I....I....I....I....I....I


     * = TOTAL DIGEST OF LTA'D COAL 401B

     O = COAL SAMPLE 400C

     . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 16

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  NA 5895.92   ***                 27-SEP-85


STEP  ANG.        INCREMENT =  345.1 COUNTS     FULL SCALE = 20704. COUNTS

               I....I....I....I....I....I....I....I....I....I....I....I....I
-11  5895.30                                          .O
-10  5895.36                                          .
 -9  5895.41                                          .
 -8  5895.47                                          .
 -7  5895.52                                         .O*
 -6  5895.58                                         . O
 -5  5895.64                                          .O
 -4  5895.69                                           .*
 -3  5895.                                            .O  *
 -2  5895.81                                          .   O     *
 -1  5895.86                                           .     O          *
  0  5895.92                                           .          O        *
  1  5895.97                                           .        O       *
  2  5896.03                                           .    O    *
  3  5896.08                                         .  O *
  4  5896.14                                         .O
  5  5896.20                                         .*
  6  5896.25                                         .O
  7  5896.31                                         *.
  8  5896.37                                          .
  9  5896.43                                         O.
 10  5896.48                                         O.
 11  5896.54                                         .
               I....I....I....I....I....I....I....I....I....I....I....I....I


     * = TOTAL DIGEST OF LTA'D COAL 401G

     O = COAL SAMPLE 400C

     . = REAGENT BLANK FOR COAL DIGESTS
```

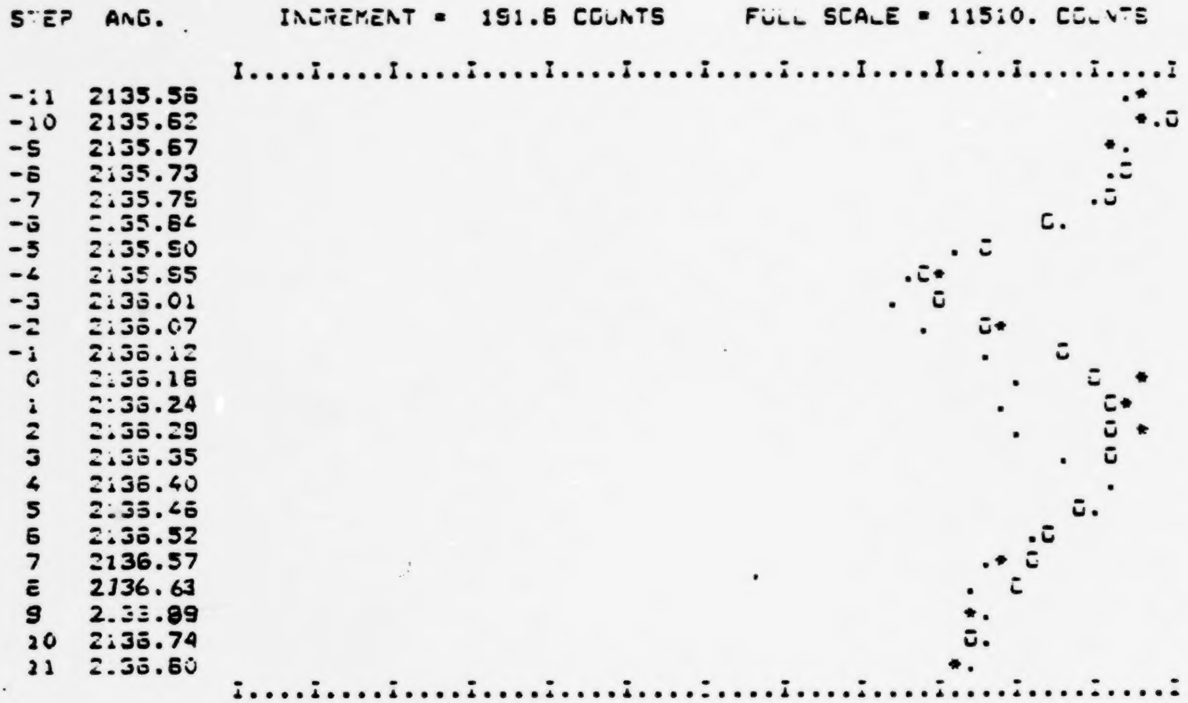
Figure 17



```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  NI 2316.04   ***               27-SEP-85



STEP  ANG.      INCREMENT =  355.3 COUNTS      FULL SCALE = 23716. COUNTS

               I....I....I....I....I....I....I....I....I....I....I....I....I
-11  2315.42                                                 .
-10  2315.48                                                  O.
-9   2315.53                                                    *.O
-8   2315.59                                                      *.
-7   2315.65                                                     *.O
-6   2315.70                                                    .
-5   2315.76                                                  O.
-4   2315.81                                                   *.
-3   2315.87                                                     .O
-2   2315.93                                                      . O
-1   2315.98                                                      .*O
 0   2316.04                                                        .*O
 1   2316.10                                                          .*O
 2   2316.15                                                            .*O
 3   2316.21                                                             *.O
 4   2316.26                                                             *.O
 5   2316.32                                                           * .
 6   2316.38                                                       * .
 7   2316.43                                                   *O.
 8   2316.49                                                 O.
 9   2316.55                                                .
 10  2316.60                                                .O
 11  2316.66                                                .O
               I....I....I....I....I....I....I....I....I....I....I....I....I


      * = TOTAL DIGEST OF LTA'D COAL 401G

      O = COAL SAMPLE 400C

      . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 18

'TOTAL' DIGEST OF LTA'D COAL SAMPLES*** P 2136.18 *** 27-SEP-85



* = TOTAL DIGEST OF LTA'D COAL 4018

G = COAL SAMPLE 400C

. = REAGENT BLANK FOR COAL DIGESTS

Figure 19

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  PB 2169.99   ***              27-SEP-85


STEP  ANG.        INCREMENT =  163.5 COUNTS      FULL SCALE = 5632.0 COUNTS

                 I....I....I....I....I....I....I....I....I....I....I....I....I
-11   2169.37                                                         .*C
-10   2169.43                                                         .*C
 -9   2169.48                                                          .*
 -8   2169.54                                                         .C
 -7   2169.60                                                          .
 -6   2169.65                                                          .*C
 -5   2169.71                                                          . * C
 -4   2169.76                                                            *.
 -3   2169.82                                                            .C*
 -2   2169.88                                                             .
 -1   2169.93                                                            . C
  0   2169.99                                                            . *C
  1   2170.05                                                             .*C
  2   2170.10                                                            C.
  3   2170.16                                                           C.
  4   2170.21                                                         C .
  5   2170.27                                                       . C
  6   2170.33                                                        .C
  7   2170.38                                                        .* C
  8   2170.44                                                         *.C
  9   2170.50                                                          .  C
 10   2170.55                                                          *.
 11   2170.61                                                           .
                 I....I....I....I....I....I....I....I....I....I....I....I....I


     * = TOTAL DIGEST OF LTA'D COAL 4016

     C = COAL SAMPLE 4003

     . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 20

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  S 1620.37   ***                 27-SEP-85



STEP  ANG.       INCREMENT =  355.7 COUNTS     FULL SCALE = 23564. COUNTS

              I....I....I....I....I....I....I....I....I....I....I....I....I
-11  1619.75     .
-10  1619.81     .
-9   1619.86     .
-8   1619.92     .
-7   1619.98     .C
-6   1620.03     .C
-5   1620.09     .C
-4   1620.14     .C
-3   1620.20     . C
-2   1620.26     .   * C
-1   1620.31      .           *    C
 0   1620.37      .                        *              C
 1   1620.43      .                               *                       C
 2   1620.48      .                         *                       C
 3   1620.54     .             *               C
 4   1620.59     .  *        C
 5   1620.65     . C
 6   1620.71     .
 7   1620.76     .
 8   1620.82     .
 9   1620.88     .
 10  1620.93     .
 11  1620.99     .
              I....I....I....I....I....I....I....I....I....I....I....I....I


    * = TOTAL DIGEST OF LTA'D COAL 4016

    C = COAL SAMPLE 4000

    . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 21

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  SB 2068.33   ***          27-SEP-83



STEP  ANG.       INCREMENT =   95.2 COUNTS      FULL SCALE = 5714.0 COUNTS

              I....I....I....I....I....I....I....I....I....I....I....I....I
-11  2067.71                                                     .C
-10  2067.77                                                    . C
-9   2067.82                                                     .   C
-8   2067.88                                                     .*    C
-7   2067.94                                                    .   *     C
-6   2067.99                                                     . *  C
-5   2068.05                                                   . *C
-4   2068.10                                                    .*C
-3   2068.16                                                     C*.
-2   2068.22                                                     . C
-1   2068.27                                                       C.
 0   2068.33                                                      .*C
 1   2068.38                                                     .*
 2   2068.44                                                   .C
 3   2068.50                                                    .C
 4   2068.55                                                    .C
 5   2066.61                                                     .C
 6   2068.67                                                     . *
 7   2068.72                                                     .* C
 8   2068.78                                                     .C*
 9   2068.84                                                    C.*
 10  2068.89                                                   *.C
 11  2068.95                                                   . C
              I....I....I....I....I....I....I....I....I....I....I....I....I


     * = TOTAL DIGEST OF LTA'D COAL 4018

     C = COAL SAMPLE 4008

     . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 22

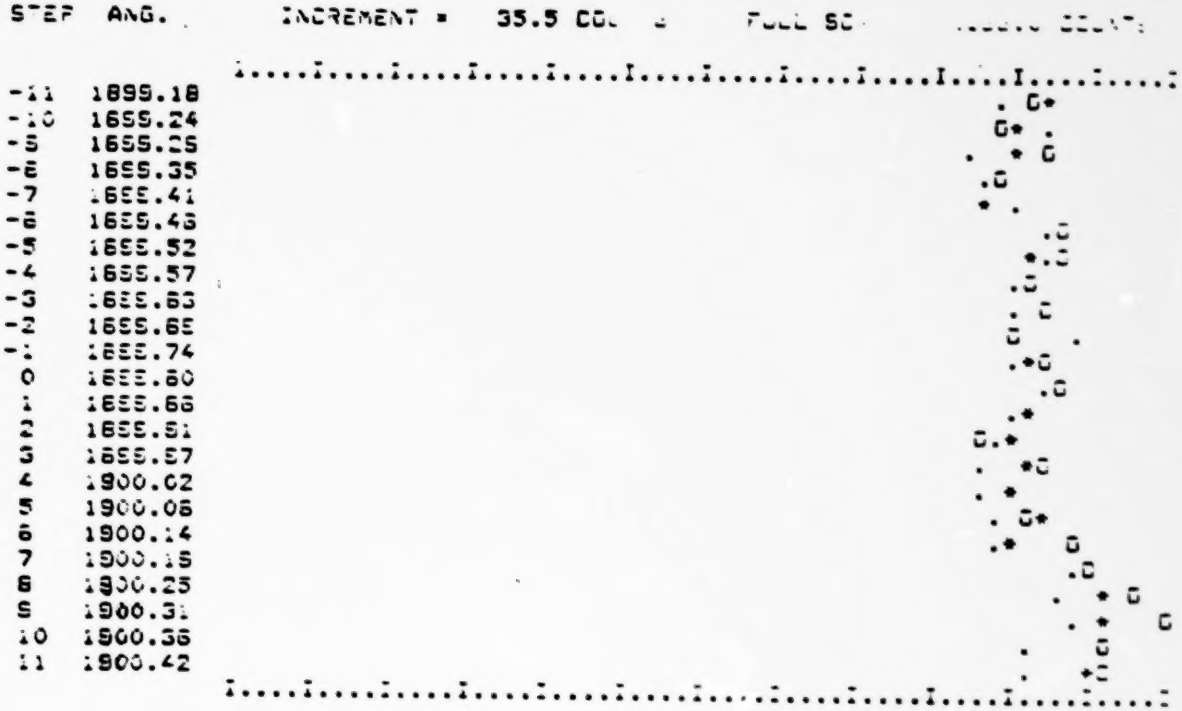
```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  SE 1960.9   ***                    27-SEP-85


STEP  ANG.        INCREMENT =   43.1 COUNTS     FULL SCALE = 2586.0 COUNTS

               I....I....I....I....I....I....I....I....I....I....I....I....I
-11  1960.28                                                            *.
-10  1960.34                                                           .  *
 -9  1960.39                                                         O .
 -8  1960.45                                                        .*O
 -7  1960.51                                                      O* .
 -6  1960.56                                                      *.    O
 -5  1960.62                                                        O.
 -4  1960.67                                                       *.
 -3  1960.73                                                       O.
 -2  1960.79                                                     . *O
 -1  1960.84                                                    .  O
  0  1960.90                                                   . * O
  1  1960.96                                                     .*   O
  2  1961.01                                                     .*      O
  3  1961.07                                                      .   *    O
  4  1961.12                                                       . *   O
  5  1961.18                                                      .  *O
  6  1961.24                                                          O.
  7  1961.29                                                        . O
  8  1961.35                                                          *O.
  9  1961.41                                                         * O.
 10  1961.46                                                         *.
 11  1961.52                                                           *.
               I....I....I....I....I....I....I....I....I....I....I....I....I


     * = TOTAL DIGEST OF LTA'D COAL 401B

     O = COAL SAMPLE 400C

     . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 23

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  SI 2881.58   ***                 27-SEP-8


STEP  ANG.          INCREMENT =  373.5 COUNTS      FULL SCALE = 22408. COUNTS

               I....I....I....I....I....I....I....I....I....I....I....I....I
-11  2880.98     .
-10  2881.03     .
 -9  2881.09     .
 -8  2881.13     .
 -7  2881.19     .
 -6  2881.24     .
 -5  2881.30     .*
 -4  2881.35     .O*
 -3  2881.41     .   O *
 -2  2881.47     .         O     *
 -1  2881.52     .                          O      *
  0  2881.58      .                                              O  *
  1  2881.64      .                                                      * O
  2  2881.69      .                                     *O
  3  2881.75     .                * O
  4  2881.80     .   *O
  5  2881.86     .O
  6  2881.92     .
  7  2881.97     .
  8  2882.03     .
  9  2882.08     .
 10  2882.14     .
 11  2882.20     .
               I....I....I....I....I....I....I....I....I....I....I....I....I


     * = TOTAL DIGEST OF LTA'D COAL 401G

     O = COAL SAMPLE 400C

     . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 24

'TOTAL' DIGEST OF LTA'D COAL SAMPLES*** SN 1899.8 *** 27-SEP-8



* = TOTAL DIGEST OF LTA'D COAL 4016

C = COAL SAMPLE 4000

. = REAGENT BLANK FOR COAL DIGESTS

Figure 25

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  SR 4215.52   ***                27-SEP-85


STEP  ANG.       INCREMENT =  147.7 COUNTS     FULL SCALE = 8860.0 COUNTS

               I....I....I....I....I....I....I....I....I....I....I....I....I
-11  4214.90     .
-10  4214.96     .
 -9  4215.01     .
 -8  4215.07     .
 -7  4215.13     .
 -6  4215.18     .*
 -5  4215.24     .*
 -4  4215.29     .  *
 -3  4215.35     .     *
 -2  4215.41     .              *
 -1  4215.46     .C                                    *
  0  4215.52     .C                                                      *
  1  4215.58     .C                                                        *
  2  4215.63     .C                                    *
  3  4215.69     .                  *
  4  4215.74     .    *
  5  4215.80     .*
  6  4215.86     .*
  7  4215.91     .
  8  4215.97     .
  9  4216.03     .
 10  4216.08     .
 11  4216.14     .
               I....I....I....I....I....I....I....I....I....I....I....I....I


     * = TOTAL DIGEST OF LTA'D COAL 4018

     C = COAL SAMPLE 400C

     . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 26

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  TI 3361.21   ***                27-SEP-85



STEP  ANG.        INCREMENT = 3729.1 COUNTS     FULL SCALE = 223744 COUNTS

                  I....I....I....I....I....I....I....I....I....I....I....I....I
-11  3360.59                 .
-10  3360.65                 .
 -9  3360.70                .
 -8  3360.76                .
 -7  3360.82               .
 -6  3360.87               .O
 -5  3360.93               .O
 -4  3360.98                .O
 -3  3361.04                .  *O
 -2  3361.10                .        O
 -1  3361.15                .                        O
  0  3361.21               .                                       * O
  1  3361.27               .                                             *    O
  2  3361.32              .                                   *     O
  3  3361.38              .                  *    O
  4  3361.43              .     *O
  5  3361.49               .O
  6  3361.55               .
  7  3361.60              .
  8  3361.66             .*
  9  3361.72             .
 10  3361.77              .
 11  3361.83               .
                  I....I....I....I....I....I....I....I....I....I....I....I....I


      * = TOTAL DIGEST OF LTA'D COAL 401G

      O = COAL SAMPLE 400C

      . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 27

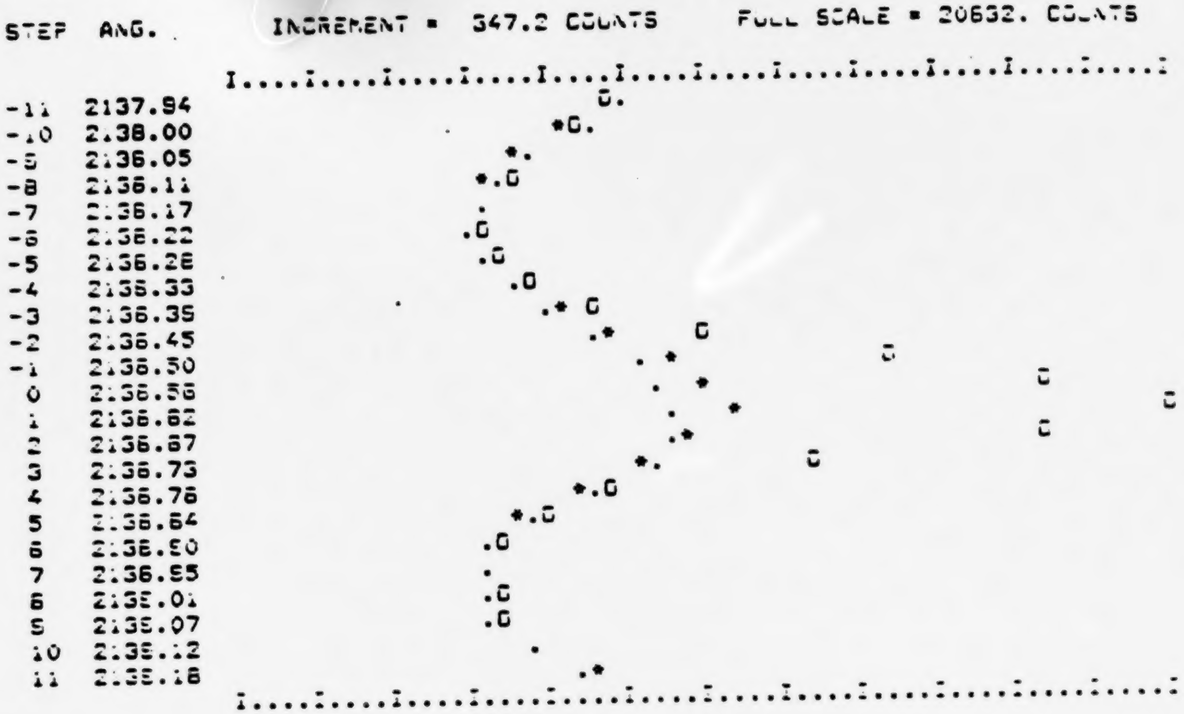
```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  TL 1906.64   ***                  27-SEP-83



STEP  ANG.        INCREMENT =   57.7 COUNTS     FULL SCALE = 3464.0 COUNTS

              I....I....I....I....I....I....I....I....I....I....I....I....I
-11  1906.02                                  C.
-10  1906.08                                   . C *
-9   1906.13                                    . C   *
-8   1906.19                                   .          C  *
-7   1906.25                                      .                  *C
-6   1906.30                                    .                    *    C
-5   1906.36                                        .           *  C
-4   1906.41                                       .    *  C
-3   1906.47                                       .C
-2   1906.53                                    .C
-1   1906.58                                   .
 0   1906.64                                 .* C
 1   1906.70                                   .
 2   1906.75                                   .*
 3   1906.81                                   .C
 4   1906.86                                   .C*
 5   1906.92                                   *. C
 6   1906.98                                     .C
 7   1907.03                                    . C
 8   1907.09                                    C.
 9   1907.15                                    .   C*
10   1907.20                                    .       C*
11   1907.26                                   .              C
              I....I....I....I....I....I....I....I....I....I....I....I....I


       * = TOTAL DIGEST OF LTA'D COAL 401G

       C = COAL SAMPLE 400C

       . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 21

```
'TOTAL' DIGEST OF LTA'D COAL SAMPLES***  W 2524.02    ***                  27-SEP-85


STEP  ANG.       INCREMENT =  567.1 COUNTS     FULL SCALE = 34024. COUNTS

              I....I....I....I....I....I....I....I....I....I....I....I....I
-11  2523.40                                                .*  C
-10  2523.46                                                 . C
-9   2523.51                                                . C
-8   2523.57                                                .*C
-7   2523.63                                                .C
-6   2523.68                                                .C
-5   2523.74                                                .C
-4   2523.79                                                .*C
-3   2523.85                                                .* C
-2   2523.91                                                . *  C
-1   2523.96                                                   *      C
 0   2524.02                                                 . *         C
 1   2524.08                                                 . *          C
 2   2524.13                                                 . *      C
 3   2524.19                                                . *  C
 4   2524.24                                                .*C
 5   2524.30                                                .C
 6   2524.36                                                 .C
 7   2524.41                                                  .
 8   2524.47                                                   .C
 9   2524.53                                                  .* C
 10  2524.58                                                  .*   C
 11  2524.64                                                  . *     C
              I....I....I....I....I....I....I....I....I....I....I....I....I


    * = TOTAL DIGEST OF LTA'D COAL 4018

    C = COAL SAMPLE 4002

    . = REAGENT BLANK FOR COAL DIGESTS
```

Figure 29

TOTAL DIGEST OF LTA'D COAL SAMPLES [illegible] 27-SEP-85



* = TOTAL DIGEST OF LTA'D COAL 401G

C = COAL SAMPLE 400C

. = REAGENT BLANK FOR COAL DIGESTS

Figure 30

APPENDIX D

SORBENT SIZE DISTRIBUTION ANALYSIS REPORTS

COMMERCIAL TESTING & ENGINEERING CO.
GENERAL OFFICES 1919 SOUTH HIGHLAND AVE. SUITE 210 B LOMBARD, ILLINOIS 60148 • (312) 953 9300

LL W. TAYLOR III
MANAGER
MIDWEST DIVISION

PLEASE ADDRESS ALL CORRESPONDENCE
16130 VAN DRUNEN RD. P.O. BO
SOUTH HOLLAND IL 6
OFFICE TEL (312) 264

ACUREX CORPORATION
P.O. Box 13109
RTP, NC 27709

ATTN: Jeff Lorrain

September 12, 1985

Sample identification
by Acurex Corp.

Kind of sample reported to us Lime

Sample taken at -----

Sample taken by Acurex Corp.

Date sampled 8/1/85

Date received 8/14/85

#1 Mercer
Huron Lime Co.
As Received
8/1/85

P.O. No. ER060912

Analysis report no. 71-11458

Mass Mode = 3.86 Microns

Mass Median= 4.43 Microns

Arithmetic Mass Mean = 7.10 Microns

FOR PLOTTING PROBABILITY ON LOG PAPER:

Percentile: 00.1% of Volume is at 46.78 Microns and Larger
Percentile: 01.0% of Volume is at 37.13 Microns and Larger
Percentile: 06.0% of Volume is at 21.33 Microns and Larger
Percentile: 22.0% of Volume is at 9.72 Microns and Larger
Percentile: 50.0% of Volume is at 4.43 Microns and Larger
Percentile: 78.0% of Volume is at 2.22 Microns and Larger
Pencentile: 94.0% of Volume is at 1.11 Microns and Larger
Pencentile: 99.0% of Volume is at .55 Microns and Larger
Pencentile: 99.9% of Volume is at .33 Microns and Larger

Density = 2.2545 g/cc

Respectfully submitted,
COMMERCIAL TESTING & ENGINEERING CO.

David W. Cox

DAVID W. COX, Manager, South Holland Laboratory

Charter Mer

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For Your Protection

DWC/ds

OVER 40 BRANCH LABORATORIES STATEGICALLY LOCATED IN PRINCIPAL COAL MINING AREAS,
TIDEWATER AND GREAT LAKES PORTS, AND RIVER LOADING FACILITIES

COMMERCIAL TESTING & ENGINEERING CO.
GENERAL OFFICES 1919 SOUTH HIGHLAND AVE SUITE 210 B LOMBARD ILLINOIS 60148 • (312) 953 9300

LL W. TAYLOR III
MANAGER
MIDWEST DIVISION

PLEASE ADDRESS ALL CORRESPONDENCE
16130 VAN DRUNEN RD P.O. BO
SOUTH HOLLAND IL 6
OFFICE TEL (312) 264

ACUREX CORPORATION
P.O. Box 13109
RTP, NC 27709

September 12, 1985

ATTN: Jeff Lorrain

Sample identification
by Acurex Corp.

Kind of sample reported to us Lime

#2 Kemikal
Kimballton, Va.
U.S. Gypsum
As Received
8/1/85

Sample taken at -----

Sample taken by Acurex Corp.

Date sampled 8/1/85

Date received 8/14/85

P.O. No. ER060912

Analysis report no. 71-11459

Mass Mode = 3.21 Microns

Mass Median= 2.79 Microns

Arithmetic Mass Mean = 3.88 Microns

FOR PLOTTING PROBABILITY ON LOG PAPER:

Percentile: 00.1% of Volume is at 29.47 Microns and Larger
Percentile: 01.0% of Volume is at 21.33 Microns and Larger
Percentile: 06.0% of Volume is at 10.18 Microns and Larger
Percentile: 22.0% of Volume is at 5.09 Microns and Larger
Percentile: 50.0% of Volume is at 2.79 Microns and Larger
Percentile: 78.0% of Volume is at 1.46 Microns and Larger
Pencentile: 94.0% of Volume is at .70 Microns and Larger
Pencentile: 99.0% of Volume is at .38 Microns and Larger
Pencentile: 99.9% of Volume is at .23 Microns and Larger

Density = 2.2729 g/cc

Respectfully submitted,
COMMERCIAL TESTING & ENGINEERING CO.

David W. Cox

DAVID W. COX, Manager, South Holland Laboratory

Charter Mem

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GENERAL OFFICES 1919 SOUTH HIGHLAND AVE SUITE 210-B. LOMBARD ILLINOIS 60148 • (312) 953-9300

[illegible] W. TAYLOR III
MANAGER
MIDWEST DIVISION

PLEASE ADDRESS ALL CORRESPONDENCE TO
16130 VAN DRUNEN RD. P.O. BOX 1
SOUTH HOLLAND IL 604
OFFICE TEL. (312) 264-1

ACUREX CORPORATION
P.O. Box 13109
RTP, NC 27709

September 12, 1985

ATTN: Jeff Lorrain

Sample identification by Acurex Corp.

Kind of sample reported to us Lime

#7 Black River
As Received
8/1/85

Sample taken at -----

Sample taken by Acurex Corp.

Date sampled 8/1/85

Date received 8/14/85

P.O. No. ER060912

Analysis report no. 71-11464

Mass Mode = 4.04 Microns

Mass Median= 4.43 Microns

Arithmetic Mass Mean = 5.53 Microns

FOR PLOTTING PROBABILITY ON LOG PAPER:

Percentile: 00.1% of Volume is at 21.33 Microns and Larger
Percentile: 01.0% of Volume is at 17.73 Microns and Larger
Percentile: 06.0% of Volume is at 12.83 Microns and Larger
Percentile: 22.0% of Volume is at 7.72 Microns and Larger
Percentile: 50.0% of Volume is at 4.43 Microns and Larger
Percentile: 78.0% of Volume is at 2.67 Microns and Larger
Pencentile: 94.0% of Volume is at 1.68 Microns and Larger
Pencentile: 99.0% of Volume is at 1.01 Microns and Larger
Pencentile: 99.9% of Volume is at .64 Microns and Larger

Density = 2.2852 g/cc

Respectfully submitted,
COMMERCIAL TESTING & ENGINEERING CO.

David W. Cox

DAVID W. COX, Manager, South Holland Laboratory

Charter Memb

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OVER 40 BRANCH LABORATORIES STATEGICALLY LOCATED IN PRINCIPAL COAL MINING AREAS,
TIDEWATER AND GREAT LAKES PORTS, AND RIVER LOADING FACILITIES

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W. TAYLOR III
MANAGER
MIDWEST DIVISION

PLEASE ADDRESS ALL CORRESPONDEN
16130 VAN DRUNEN RD P.O. B
SOUTH HOLLAND IL
OFFICE TEL (312) 264

ACUREX CORPORATION
P.O. Box 13109
RTP, NC 27709

ATTN: Jeff Lorrain

September 12, 1985

Sample identification
by Acurex Corp.

#6 Marblehead
As Received
8/1/85

Kind of sample reported to us Lime

Sample taken at -----

Sample taken by Acurex Corp.

Date sampled 8/1/85

Date received 8/14/85

P.O. No. ER060912

Analysis report no 71-11463

Mass Mode = 5.33 Microns

Mass Median= 5.33 Microns

Arithmetic Mass Mean = 7.79 Microns

FOR PLOTTING PROBABILITY ON LOG PAPER:

Percentile: 00.1% of Volume is at 56.28 Microns and Larger
Percentile: 01.0% of Volume is at 40.73 Microns and Larger
Percentile: 06.0% of Volume is at 20.36 Microns and Larger
Percentile: 22.0% of Volume is at 10.66 Microns and Larger
Percentile: 50.0% of Volume is at 5.33 Microns and Larger
Percentile: 78.0% of Volume is at 2.79 Microns and Larger
Pencentile: 94.0% of Volume is at 1.53 Microns and Larger
Pencentile: 99.0% of Volume is at .80 Microns and Larger
Pencentile: 99.9% of Volume is at .44 Microns and Larger

Density = 2.2158 g/cc

Respectfully submitted,
COMMERCIAL TESTING & ENGINEERING CO.

David W. Cox

DAVID W. COX, Manager, South Holland Laboratory

Charter M

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LL[illegible] W. TAYLOR III
MANAGER
MIDWEST DIVISION

PLEASE ADDRESS ALL CORRESPONDENCE
16130 VAN DRUNEN RD P O BO
SOUTH HOLLAND IL 6
OFFICE TEL (312) 264

ACUREX CORPORATION
P.O. Box 13109
RTP, NC 27709

September 12, 1985

ATTN: Jeff Lorrain

Sample identification by Acurex Corp.

Kind of sample reported to us: Lime

#5 Detroit
Lime Calcite
U.S. Gypsum
As Received
8/1/85

Sample taken at: -----

Sample taken by: Acurex Corp.

Date sampled: 8/1/85

Date received: 8/14/85

P.O. No. ER060912

Analysis report no. 71-11462

Mass Mode = 8.08 Microns

Mass Median= 7.37 Microns

Arithmetic Mass Mean = 8.33 Microns

FOR PLOTTING PROBABILITY ON LOG PAPER:

Percentile: 00.1% of Volume is at 28.14 Microns and Larger
Percentile: 01.0% of Volume is at 23.39 Microns and Larger
Percentile: 06.0% of Volume is at 17.73 Microns and Larger
Percentile: 22.0% of Volume is at 11.70 Microns and Larger
Percentile: 50.0% of Volume is at 7.37 Microns and Larger
Percentile: 78.0% of Volume is at 4.43 Microns and Larger
Pencentile: 94.0% of Volume is at 2.12 Microns and Larger
Pencentile: 99.0% of Volume is at 1.01 Microns and Larger
Pencentile: 99.9% of Volume is at .55 Microns and Larger

Density = 2.2066 g/cc

Respectfully submitted,
COMMERCIAL TESTING & ENGINEERING CO.

David W. Cox

DAVID W. COX, Manager, South Holland Laboratory

Charter M

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DWC/ds

COMMERCIAL TESTING & ENGINEERING CO.
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LL W. TAYLOR III
MANAGER
MIDWEST DIVISION

PLEASE ADDRESS ALL CORRESPONDENCE
16130 VAN DRUNEN RD P O BOX
SOUTH HOLLAND IL 6
OFFICE TEL (312) 264

ACUREX CORPORATION
P.O. Box 13109
RTP, NC 27709

ATTN: Jeff Lorrain

September 12, 1985

Sample identification by Acurex Corp.

#4 Ivory
Dolomitic
U.S. Gypsum
As Received
8/1/85

Kind of sample reported to us Lime

Sample taken at -----

Sample taken by Acurex Corp.

Date sampled 8/1/85

Date received 8/14/85

P.O. No. ER060912

Analysis report no. 71-11461

Mass Mode = 2.17 Microns

Mass Median= 4.59 Microns

Arithmetic Mass Mean = 13.72 Microns

FOR PLOTTING PROBABILITY ON LOG PAPER:

Percentile: 00.1% of Volume is at 92.49 Microns and Larger
Percentile: 01.0% of Volume is at 77.78 Microns and Larger
Percentile: 06.0% of Volume is at 51.91 Microns and Larger
Percentile: 22.0% of Volume is at 21.83 Microns and Larger
Percentile: 50.0% of Volume is at 4.59 Microns and Larger
Percentile: 78.0% of Volume is at 1.53 Microns and Larger
Pencentile: 94.0% of Volume is at .61 Microns and Larger
Pencentile: 99.0% of Volume is at .30 Microns and Larger
Pencentile: 99.9% of Volume is at .21 Microns and Larger

Density = 2.2872 g/cc

Respectfully submitted
COMMERCIAL TESTING & ENGINEERING CO.

David W. Cox

DAVID W. COX, Manager, South Holland Laboratory

Charter Me

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For Your Protection

DWC/ds

OVER 40 BRANCH LABORATORIES STATEGICALLY LOCATED IN PRINCIPAL COAL MINING AREAS,
TIDEWATER AND GREAT LAKES PORTS, AND RIVER LOADING FACILITIES

COMMERCIAL TESTING & ENGINEERING CO.
GENERAL OFFICES 1919 SOUTH HIGHLAND AVE SUITE 210 B LOMBARD, ILLINOIS 60148 • (312) 953-9300

[illegible] W. TAYLOR III
MANAGER
MIDWEST DIVISION

PLEASE ADDRESS ALL CORRESPONDENC
16130 VAN DRUNEN RD P.O. BC
SOUTH HOLLAND IL 6
OFFICE TEL (312) 264

ACUREX CORPORATION
P.O. Box 13109
RTP, NC 27709

September 12, 1985

ATTN: Jeff Lorrain

Sample identification
by Acurex Corp.

Kind of sample reported to us Lime

#3 Remidol
Dolomitic
Type N
U.S. Gypsum
As Received
8/1/85

Sample taken at -----

Sample taken by Acurex Corp.

Date sampled 8/1/85

Date received 8/14/85

P.O. No. ER060912

Analysis report no. 71-11460

Mass Mode = 33.85 Microns

Mass Median= 12.83 Microns

Arithmetic Mass Mean = 19.71 Microns

FOR PLOTTING PROBABILITY ON LOG PAPER:

Percentile: 00.1% of Volume is at 85.31 Microns and Larger
Percentile: 01.0% of Volume is at 77.78 Microns and Larger
Percentile: 06.0% of Volume is at 58.94 Microns and Larger
Percentile: 22.0% of Volume is at 33.85 Microns and Larger
Percentile: 50.0% of Volume is at 12.83 Microns and Larger
Percentile: 78.0% of Volume is at 2.22 Microns and Larger
Pencentile: 94.0% of Volume is at .96 Microns and Larger
Pencentile: 99.0% of Volume is at .55 Microns and Larger
Pencentile: 99.9% of Volume is at .42 Microns and Larger

Density = 2.3979 g/cc

Respectfully submitted.
COMMERCIAL TESTING & ENGINEERING CO.

David W. Cox

DAVID W. COX, Manager, South Holland Laboratory

Charter Me

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DWC/ds

OVER 40 BRANCH LABORATORIES STATEGICALLY LOCATED IN PRINCIPAL COAL MINING AREAS,
TIDEWATER AND GREAT LAKES PORTS AND RIVER LOADING FACILITIES

APPENDIX E

SORBENT ELEMENTAL ANALYSIS REPORTS

Sample Inventory (for elemental Analysis)

Sample I.D.	Elements to be Determined
Round Rock Limestone	Ca,Mg,Al,Si,K,Na,Fe,Mn,Ba,Sr,Ti,P,Cl,S
Clifton Limestone	↓
Genstar Giantsville Dolomite	
Maysville Limestone	
Longview Limestone	
Acurex Dolomite	
El Dorado Limestone	
Vicron, Mass.	
Vicron, Ca.	
Detroit Limestone	
Mississippi Limestone	
Ash Grove Limestone	
Genstar Nelson Limestone	
Genstar Henderson Limestone	
U.S. Gypsum, Genda, Ohio, Dolomitic Limestone	
U.S. Gypsum, Texas, Limestone	
U.S. Gypsum, Texas Dolomitic Limestone	
U.S. Gypsum, Kimballton, Va.	
Presque Isle Limestone	
Inland Limestone	
Camden Limestone	
Thorton Dolomite	
Pleasant Gap Limestone	
River Rouge	
Tyrochter Dolomite	
Ivory as Rec'd 8/8/85	
Black Riber as Rec'd 8/8/85	
Marblehead as Rec'd 8/8/85	
MERCER as Rec'd 8/8/85	
Kemidol as Rec'd 8/8/85	
Detroid as Rec'd 8/8/85	
Kemikal as Rec'd 8/8/85	Ca,Mg,Al,Si,K,Na,Fe,Mn,Ba,Sr,Ti,P,Cl,S
Hydrated Whitestone	Si,Al,Fe,Mn,Na
Hydrated Tate 6/26/85	Si,Al,Fe,Mn,Na
$CaCO_3$	Na
$Ca(OH)_2$	Na

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[illegible]
MANAGER
INSTRUMENTAL ANALYSIS DIVISION

PLEASE ADDRESS ALL CORRESPONDENCE
490 ORCHARD ST. GOLDEN, CO
OFFICE TEL (303) 27[illegible]

To: Mr. Jeff Lorrain
Acurex Corporation
P. O. Box 13109
Research Triangle Park, NC 27709

Date: August 29, 1985
IAD #97-T334-116-36
Received: 08/14/85

Material: Limestone and Lime Hydrate Samples

Analysis: Elemental analysis of SiO_2, Al_2O_3, TiO_2, Fe_2O_3, CaO, MgO, K_2O, Na_2O, SO_3, P_2O_5, SrO, BaO, Mn_3O_4 and Cl.

Procedures: A representative portion of each sample was ignited at 1000°C. An accurately weighed portion of ignited sample was fused, with lithium tetraborate/lithium carbonate (90/10), to form a glass disk suitable for analysis by X-ray fluorescence spectrometry. The Chlorine samples were dissolved in a 5% HNO_3 solution and analyzed by ion selective electrode.

Results: Loss on Ignition at 1000°C and Elemental Analysis are reported in weight percent (Wt.%) on an ignited basis, except for Chlorine which is reported in microgram per gram (µg/g) on an as received basis. Moistures are reported on an as received basis.

If you have any questions regarding this analysis, please call.

Norman Perkins
X-ray Supervisor

Charter Member

OVER 40 BRANCH LABORATORIES STRATEGICALLY LOCATED IN PRINCIPAL COAL MINING AREAS,
TIDEWATER AND GREAT LAKES PORTS, AND RIVER LOADING FACILITIES

Parameter	Round Rock Limestone	Clifton Limestone	Gemstar Grantsville Dolomite	Maysville Limestone	Longview Limestone
Moisture	0.02	0.02	0.10	0.06	0.03
Loss on Ignition	42.62	42.99	28.08	43.56	43.19
Silica, SiO_2	0.27	0.14	1.91	1.79	0.45
Alumina, Al_2O_3	0.16	0.08	0.29	0.38	0.19
Titania, TiO_2	0.00	0.00	0.01	0.00	0.00
Ferric oxide, Fe_2O_3	0.00	0.28	0.47	0.13	0.00
Calcium oxide, CaO	98.19	98.30	57.01	88.03	96.56
Magnesia, MgC	1.15	0.99	38.42	9.36	2.41
Potassium oxide, K_2O	0.03	0.02	0.12	0.12	0.09
Sodium oxide, Na_2O	0.00	0.00	0.00	0.00	0.00
Sulfur trioxide, SO_3	0.12	0.12	0.09	0.16	0.24
Phosphorus pentoxide, P_2O_5	0.00	0.00	0.00	0.00	0.00
Strontium oxide, SrO	0.05	0.06	0.00	0.00	0.02
Barium oxide, BaO	0.02	0.00	0.02	0.02	0.04
Manganese oxide, Mn_3O_4	0.01	0.01	0.04	0.01	0.00
Chloride, Cl (ug/g)	≤180	≤180	≤180	≤180	≤180
SUM	100.00	100.00	100.00	100.00	100.00

Parameter	Acurex Dolomite	El Dorado Limestone	Vicron Mass	Vicron Ca	Detroit Limestone
Moisture	0.00	0.00	0.00	0.01	0.02
Loss On Ignition	46.33	42.49	41.65	43.05	43.62
Silica, SiO_2	0.32	0.21	1.99	0.00	0.14
Alumina, Al_2O_3	0.00	0.00	0.30	0.00	0.00
Titania, TiO_2	0.00	0.00	0.00	0.00	0.00
Ferric oxide, Fe_2O_3	0.25	0.00	0.00	0.00	0.00
Calcium oxide, CaO	50.71	97.52	95.70	97.44	97.54
Magnesia, MgO	42.53	2.00	1.47	2.38	1.63
Potassium oxide, K_2O	0.02	0.04	0.27	0.03	0.03
Sodium oxide, Na_2O	0.00	0.00	0.00	0.00	0.28
Sulfur trioxide, SO_3	0.12	0.13	0.22	0.13	0.33
Phosphorus pentoxide, P_2O_5	0.01	0.02	0.01	0.01	0.00
Strontium oxide, SrO	0.00	0.06	0.04	0.00	0.04
Barium oxide, BaO	0.02	0.01	0.00	0.00	0.00
Manganese oxide, Mn_3O_4	0.07	0.01	0.00	0.01	0.01
Chloride, Cl (ug/g)	≤180	≤180	≤180	≤180	≤180
SUM	94.05	100.00	100.00	100.00	100.00

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Parameter	Mississippi Limestone	Ash Grove Limestone	Gemstar Nelson Limestone	Gemstar Henderson Limestone	US Gypsu Genoa,Chi Dolomite Limestor
Moisture	0.04	0.04	0.01	0.00	0.00
Loss on Ignition	43.76	45.15	42.47	26.09	46.30
Silica, SiO_2	0.82	0.20	0.16	0.00	0.02
Alumina, Al_2O_3	0.00	0.00	0.00	0.00	0.00
Titania, TiO_2	0.00	0.00	0.00	0.00	0.00
Ferric oxide, Fe_2O_3	0.00	0.00	0.00	0.00	0.08
Calcium oxide, CaO	96.93	98.61	81.01	60.15	56.24
Magnesia, MgO	2.04	1.00	0.79	35.48	37.63
Potassium oxide, K_2O	0.04	0.02	0.02	0.02	0.02
Sodium oxide, Na_2O	0.00	0.00	0.00	0.03	0.00
Sulfur trioxide, SO_3	0.17	0.12	0.13	0.21	0.64
Phosphorus pentoxide, P_2O_5	0.00	0.00	0.00	0.00	0.00
Strontium oxide, SrO	0.00	0.03	0.00	0.00	1.04
Barium oxide, BaO	0.00	0.00	0.01	0.01	0.02
Manganese oxide, Mn_3O_4	0.00	0.02	0.01	0.04	0.00
Chloride, Cl (ug/g)	≤180	≤180	≤180	870	760
SUM	100.00	100.00	82.13	96.00	95.72

Parameter	US Gypsum TX Limestone	US Gypsum Dolomite Limestone	US Gypsum Kimballton VA	Presque Isle Limestone	Inland Limestone
Moisture	0.01	0.05	0.10	0.00	0.01
Loss On Ignition	42.75	50.51	42.91	43.55	43.40
Silica, SiO_2	0.21	1.82	1.53	0.62	0.56
Alumina, Al_2O_3	0.02	0.44	0.34	0.13	0.07
Titania, TiO_2	0.00	0.00	0.00	0.00	0.00
Ferric oxide, Fe_2O_3	0.00	0.25	0.08	0.15	0.07
Calcium oxide, CaO	95.87	55.55	95.99	97.61	96.91
Magnesia, MgO	0.70	36.23	1.78	1.18	0.00
Potassium oxide, K_2O	0.02	0.09	0.07	0.05	0.04
Sodium oxide, Na_2O	0.16	0.00	0.00	0.00	0.00
Sulfur trioxide, SO_3	0.12	0.10	0.19	0.23	0.26
Phosphorus pentoxide, P_2O_5	0.01	0.00	0.01	0.00	0.00
Strontium oxide, SrO	0.03	0.00	0.03	0.00	0.00
Barium oxide, BaO	0.01	0.02	0.03	0.01	0.00
Manganese oxide, Mn_3O_4	0.00	0.01	0.01	0.02	0.02
Chloride, Cl (ug/g)	≤180	350	≤180	310	300
SUM	97.15	94.53	100.06	100.02	97.94

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97-T234-116-36
age 4

Parameter	Camden Limestone	Thorton Dolomite	Pleasant Gap Limestone	River Rouge	Tyrochte Dolomite
Moisture	0.00	0.08	0.01	0.01	0.05
Loss on Ignition	42.91	46.95	42.64	43.93	45.00
Silica, SiO_2	0.00	0.85	2.28	0.26	4.13
Alumina, Al_2O_3	0.00	0.21	0.78	0.03	1.63
Titania, TiO_2	0.00	0.00	0.02	0.00	0.04
Ferric oxide, Fe_2O_3	0.00	1.27	0.11	0.04	1.74
Calcium oxide, CaO	97.21	58.04	93.46	83.30	51.21
Magnesia, MgO	0.65	38.72	1.45	0.00	38.23
Potassium oxide, K_2O	0.03	0.03	0.17	0.03	0.19
Sodium oxide, Na_2O	0.07	0.00	0.03	0.00	0.00
Sulfur trioxide, SO_3	0.12	0.26	0.28	0.26	0.81
Phosphorus pentoxide, P_2O_5	0.01	0.00	0.01	0.01	0.00
Strontium oxide, SrO	0.03	0.00	0.06	0.00	0.03
Barium oxide, BaO	0.00	0.01	0.00	0.02	0.00
Manganese oxide, Mn_3O_4	0.01	0.02	0.00	0.01	0.02
Chloride, Cl (ug/g)	≤180	1450	≤180	410	600
SUM	98.13	99.49	98.65	83.98	98.06

Parameter	Ivory As Rec. 8/8/85	Black River As Rec. 8/8/85	Marblehead As Rec. 8/8/85	Mercer As Rec. 8/8/85	Kemidol As Rec. 8/8/85
Moisture	0.00	0.22	0.55	0.36	0.13
Loss On Ignition	26.40	43.08	24.45	24.07	23.49
Silica, SiO_2	0.10	1.55	1.38	1.13	0.12
Alumina, Al_2O_3	0.00	0.53	0.32	0.38	0.00
Titania, TiO_2	0.00	0.00	0.00	0.00	0.00
Ferric oxide, Fe_2O_3	0.00	0.00	0.33	0.50	0.00
Calcium oxide, CaO	57.41	97.02	65.22	96.13	57.15
Magnesia, MgO	39.17	0.58	0.81	1.56	39.19
Potassium oxide, K_2O	0.02	0.08	0.02	0.03	0.02
Sodium oxide, Na_2O	0.00	0.00	0.00	0.00	0.00
Sulfur trioxide, SO_3	0.85	0.18	0.24	0.17	0.82
Phosphorus pentoxide, P_2O_5	0.00	0.01	0.01	0.01	0.00
Strontium oxide, SrO	0.96	0.04	0.03	0.06	0.81
Barium oxide, BaO	0.04	0.01	0.00	0.01	0.03
Manganese oxide, Mn_3O_4	0.02	0.00	0.01	0.02	0.01
Chloride, Cl (ug/g)	820	≤180	≤180	≤180	830
SUM	98.59	100.00	68.37	100.00	98.17

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Parameter	Detroid As Rec. 8/8/85	Kemikal As Rec. 8/8/85	Hydrated Whitestone	Hydrated Tate 6/26/85	$CaCO_3$
Moisture	0.51	0.46	0.44	0.23	0.00
Loss on Ignition	24.29	24.02	21.25	23.35	42.78
Silica, SiO_2	0.59	1.57	4.59	2.40	
Alumina, Al_2O_3	0.13	0.51	1.26	0.49	
Titania, TiO_2	0.00	0.01			
Ferric oxide, Fe_2O_3	0.16	0.00	1.20	0.15	
Calcium oxide, CaO	76.80	95.61			99.95
Magnesia, MgO	0.00	1.90			
Potassium oxide, K_2O	0.02	0.07			
Sodium oxide, Na_2O	0.00	0.15	0.00	0.00	0.00
Sulfur trioxide, SO_3	0.22	0.09			
Phosphorus pentoxide, P_2O_5	0.01	0.01			
Strontium oxide, SrO	0.03	0.06			
Barium oxide, BaO	0.00	0.02			
Manganese oxide, Mn_3O_4	0.02	0.00	0.04	0.02	
Chloride, Cl (ug/g)	$\leq$180	$\leq$180			
SUM	77.98	100.00			

Parameter	$Ca(OH)_2$
Moisture	0.00
Loss On Ignition	24.60
Silica, SiO_2	0.61
Calcium oxide, CaO	94.99
Sodium oxide, Na_2O	0.00

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C ALD T SKAR
MANAGER
INSTRUMENTAL ANALYSIS DIVISION

PLEASE ADDRESS ALL CORRESPONDENC
490 ORCHARD ST GOLDEN CO
OFFICE TEL (303) 278

To: Mr. Jeff Lorrain
Acurex Corporation
P. O. Box 13109
Research Triangle Park, NC 27709

Date: September 25, 1985
IAD #97-T334-116-36
Received: 08/14/85
Amended Report

Material: Limestone and Lime Hydrate Samples

Analysis: Elemental analysis of SiO_2, Al_2O, TiO_2, Fe_2O_3, CaO, MgO, K_2O, Na_2O, SO_3, P_2O_5, SrO, BaO, Mn_3O_4 and Cl.

Procedures: A representative portion of each sample was ignited at 1000°C, ground, and then reignited. An accurately weighed portion of ignited sample was fused with lithium tetraborate/lithium carbonate (90/10), to form a glass disk suitable for analysis by X-ray fluorescence spectrometry. The Chlorine samples were dissolved in a 5% HNO_3 solution and analyzed by ion selective electrode.

Results: Loss on Ignition at 1000°C and Elemental Analysis are reported in weight percent (Wt.%) on an ignited basis, except for Chlorine which is reported in micrograms per gram (ug/g) on an as received basis. Moistures are reported on an as received basis.

If you have any questions regarding this analysis, please call.

Norman Perkins
Supervisor
X-Ray Fluorescence Section

Charter Member

OVER 40 BRANCH LABORATORIES STRATEGICALLY LOCATED IN PRINCIPAL COAL MINING AREAS,
TIDEWATER AND GREAT LAKES PORTS, AND RIVER LOADING FACILITIES

Parameter	Gemstar Nelson Limestone	River Rogue	Marblehead As Rec'd 8/8/85	Detroit As Rec'd 8/8/85
Moisture	0.01	0.01	0.55	0.51
Loss on Ignition	42.47	43.93	24.07	24.29
Silica, SiO_2	0.83	0.26	1.20	1.74
Alumina, Al_2O_3	0.54	0.41	0.90	1.13
Titania, TiO_2	0.00	0.00	0.00	0.02
Ferric oxide, Fe_2O_3	4.21	0.05	0.41	0.69
Calcium oxide, CaO	93.39	95.11	95.74	92.88
Magnesia, MgO	0.80	1.39	1.27	1.72
Potassium oxide, K_2O	0.03	0.03	0.04	0.08
Sodium oxide, Na_2O	0.00	0.00	0.12	0.00
Sulfur trioxide, SO_3	0.16	0.35	0.24	0.50
Phosphorus pentoxide, P_2O_5	0.01	0.01	0.00	0.01
Strontium oxide, SrO	0.00	0.00	0.02	0.04
Barium oxide, BaO	0.02	0.01	0.02	0.02
Manganese oxide, Mn_3O_4	0.01	0.01	0.01	0.01
Chloride, Cl (ug/g)	≤180	≤180	≤180	
SUM	100.00	98.20	99.97	98.84

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